Multi-Area Quality Assurance Project Plan

12th Street Landfill Site (Operable Unit No. 4), Kalamazoo River (Operable Unit No. 5), and Plainwell Mill Site (Operable Unit No. 7)

Allied Paper, Inc./Portage Creek/
Kalamazoo River Superfund Site
Allegan County, Michigan

Revision 02, Addendum 03
November 2008

Submitted in connection with Phase 2 of Addendum No. 1 (Test Pits) and the Plainwell Mill RI/FS Workplan (Phase 1 Initial Groundwater Assessment)

Prepared by RMT, Inc.
on behalf of Weyerhaeuser Company

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Table of Contents

Acronyms and Abbreviations v

1. Introduction 1-1
   - 1.1 Development of the Multi-Area QAPP 1-2
   - 1.2 Project Setting 1-2
   - 1.3 Applicability of the Multi-Area QAPP 1-3

2. Scope of Activities 2-1
   - 2.1 Emergency Response Activities at the 12th Street Landfill (Operable Unit No. 4) 2-1
     - 2.1.1 Former Powerhouse Discharge Channel 2-2
     - 2.1.2 Eastern Bank of the Landfill 2-2
   - 2.2 Emergency Response Activities at the Former Plainwell Mill Banks (Operable Unit No. 5) 2-3
   - 2.3 Predesign Investigations for the Remedial Design for the 12th Street Landfill (Operable Unit No. 4) 2-4
     - 2.3.1 Extent of Paper Residuals Beyond Landfill Footprint 2-5
     - 2.3.2 Grading Design 2-6
     - 2.3.3 Landfill Gas Management 2-7
   - 2.4 Phase 2 of Addendum No. 1 Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment (Operable Unit No. 7) 2-7
     - 2.4.1 Phase 2 of Addendum No. 1 2-7
     - 2.4.2 Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment 2-8

3. Document Overview 3-1

4. References 4-1

List of Figures

Figure 1-1 Site Location Map
Figure 1-2 Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Figure 2-1 Historical Sampling Data for the Former Powerhouse Discharge Channel
Figure 2-2 Proposed Wetland, Woodland, and Asphalt Plant Area Investigations
Figure 2-3 Proposed Landfill Predesign Investigations

List of Attachments

Attachment 1-1 Laboratory Accuracy and Precision Objectives
Attachment 1-2 Surrogate Compound Recovery Limits
Attachment 1-3 Sample Chain-of-Custody Record
Attachment 1-4 Laboratory Quality Assurance Manual
Attachment 1-5 Laboratory SOPs
Attachment 1-6 Laboratory Policies and Guidelines

List of Worksheets

WORKSHEET	DATE OF LAST REVISION	TITLE
QAPP Worksheet #1	November 2008	Title and Approval Page
QAPP Worksheet #2	November 2008	Identifying Information
QAPP Worksheet #3	April 2008	Distribution List
QAPP Worksheet #4-1	April 2008	Project Personnel Sign-Off Sheet (RMT)
QAPP Worksheet #4-2	September 2007	Project Personnel Sign-Off Sheet (WATS)
QAPP Worksheet #5-1	September 2007	Project Organizational Chart (Emergency Response Activities for the 12th Street Landfill and the Plainwell Mill Banks)
QAPP Worksheet #5-2	April 2008	Project Organization Chart (Predesign Investigations for the Remedial Design for the 12th Street Landfill)
QAPP Worksheet #5-3	November 2008	Project Organization Chart (Phase 2 of Addendum No. 1 [Test Pits] and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)
QAPP Worksheet #6-1	September 2007	Communication Pathways (Emergency Response Activities for the 12th Street Landfill and the Plainwell Mill Banks)
QAPP Worksheet #6-2	April 2008	Communication Pathways (Predesign Investigations for the Remedial Design for the 12th Street Landfill)
QAPP Worksheet #6-3	November 2008	Communication Pathways (Phase 2 of Addendum No. 1 [Test Pits] and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)
QAPP Worksheet #7	April 2008	Personnel Responsibilities and Qualifications
QAPP Worksheet #8	November 2008	Special Personnel Training Requirements
QAPP Worksheet #9-1	June 2007	Project Scoping Session Participants Sheet (May 8, 2007)
QAPP Worksheet #9-2	June 2007	Project Scoping Session Participants Sheet (June 6, 2007)
QAPP Worksheet #9-3	April 2008	Project Scoping Session Participants Sheet (December 7, 2007)
QAPP Worksheet #9-4	April 2008	Project Scoping Session Participants Sheet (January 9, 2008)
QAPP Worksheet #9-5	November 2008	Project Scoping Session Participants Sheet (June, 2008)
QAPP Worksheet #10-1	June 2007	Problem Definition - DQOs (Emergency Response Activities in the Former Powerhouse Discharge Channel)
QAPP Worksheet #10-2	September 2007	Problem Definition - DQOs (Emergency Response on the Plainwell Mill Banks)
QAPP Worksheet #10-3	April 2008	Problem Definition - DQOs (Predesign Investigations for the Remedial Design for the 12th Street Landfill)
QAPP Worksheet #10-4	November 2008	Problem Definition - DQOs (Phase 2 of Addendum No. 1 [Test Pits] and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)
QAPP Worksheet #11-1	September 2007	Project Quality Objectives/Systematic Planning Process Statements (Emergency Response Activities in the Former Powerhouse Discharge Channel)

WORKSHEET	DATE OF LAST REVISION	TITLE
QAPP Worksheet #11-2	September 2007	Project Quality Objectives/Systematic Planning Process Statements (Emergency Response on the Plainwell Mill Banks)
QAPP Worksheet #11-3	April 2008	Project Quality Objectives/Systematic Planning Process Statements (Predesign Investigations for the Remedial Design for the 12th Street Landfill)
QAPP Worksheet #11-4	November 2008	Project Quality Objectives/Systematic Planning Process Statements (Phase 2 of Addendum No. 1 [Test Pits] and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)
QAPP Worksheet #12-1	June 2007	Measurement Performance Criteria (PCBs - Surface Water)
QAPP Worksheet #12-2	June 2007	Measurement Performance Criteria (Wet Chemistry - Surface Water)
QAPP Worksheet #12-3	June 2007	Measurement Performance Criteria (PCBs - Soil/Sediment)
QAPP Worksheet #12-4	November 2008	Measurement Performance Criteria (PCBs – Free Product)
QAPP Worksheet #12-5	November 2008	Measurement Performance Criteria (TAL Metals – Groundwater)
QAPP Worksheet #12-6	November 2008	Measurement Performance Criteria (TAL Metals – Soil)
QAPP Worksheet #12-7	November 2008	Measurement Performance Criteria (Cyanide – Groundwater)
QAPP Worksheet #12-8	November 2008	Measurement Performance Criteria (Low-Level Mercury – Groundwater)
QAPP Worksheet #12-9	November 2008	Measurement Performance Criteria (SVOCs – Groundwater)
QAPP Worksheet #12-10	November 2008	Measurement Performance Criteria (SVOCs – Soil)
QAPP Worksheet #12-11	November 2008	Measurement Performance Criteria (VOCs – Groundwater)
QAPP Worksheet #12-12	November 2008	Measurement Performance Criteria (VOCs – Soil)
QAPP Worksheet #12-13	November 2008	Measurement Performance Criteria (DRO and GRO – Soil)
QAPP Worksheet #13-1	April 2008	Secondary Data Criteria and Limitations (12th Street Landfill)
QAPP Worksheet #13-2	November 2008	Secondary Data Criteria and Limitations (Plainwell Mill)
QAPP Worksheet #14	November 2008	Summary of Project Tasks
QAPP Worksheet #15-1	June 2007	Reference Limits and Evaluation (Surface Water)
QAPP Worksheet #15-2	November 2008	Reference Limits and Evaluation (Soil/Sediment)
QAPP Worksheet #15-3	November 2008	Reference Limits and Evaluation (Groundwater)
QAPP Worksheet #15-4	November 2008	Reference Limits and Evaluation (Free Product)
QAPP Worksheet #16-1	June 2007	Project Schedule/Timeline (Emergency Response Activities in the Former Powerhouse Discharge Channel)
QAPP Worksheet #16-2	September 2007	Project Schedule/Timeline (Emergency Response Activities on the Plainwell Mill Banks)
QAPP Worksheet #16-3	April 2008	Project Schedule/Timeline (Predesign Investigations for the Remedial Design for the 12th Street Landfill)
QAPP Worksheet #16-4	November 2008	Project Schedule/Timeline (Phase 2 of Addendum No. 1 [Test Pits] and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)
QAPP Worksheet #17-1	June 2007	Sampling Design and Rationale (Former Powerhouse Discharge Channel)
QAPP Worksheet #17-2	September 2007	Sampling Design and Rationale (Emergency Response on the Plainwell Mill Banks)
QAPP Worksheet #17-3	April 2008	Sampling Design and Rationale (Predesign Investigations for the Remedial Design For the 12th Street Landfill)
QAPP Worksheet #17-4	November 2008	Sampling Design and Rationale (Phase 2 of Addendum No. 1 [Test Pits] and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)
QAPP Worksheet #18-1	September 2007	Sampling Locations and Methods/SOP Requirements (Emergency Response Activities)
QAPP Worksheet #18-2	April 2008	Sampling Locations and Methods/SOP Requirements (Predesign Investigations for the Remedial Design For the 12th Street Landfill)

WORKSHEET	DATE OF LAST REVISION	TITLE
QAPP Worksheet #18-3	November 2008	Sampling Locations and Methods/SOP Requirements (Phase 2 of Addendum No. 1 [Test Pits] and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)
QAPP Worksheet #19	November 2008	Analytical SOP Requirements
QAPP Worksheet #20	November 2008	Field Quality Control Sample Summary
QAPP Worksheet #21	April 2008	Project Sampling SOP Reference
QAPP Worksheet #22	November 2008	Field Equipment Calibration, Maintenance, Testing, and Inspection
QAPP Worksheet #23	November 2008	Analytical SOP References
QAPP Worksheet #24	November 2008	Analytical Instrument Calibration
QAPP Worksheet #25	November 2008	Analytical Instrument and Equipment Maintenance, Testing, and Inspection
QAPP Worksheet #26	June 2007	Sample Handling System (WATS)
QAPP Worksheet #27	June 2007	Sample Custody Requirements
QAPP Worksheet #28-1	June 2007	QC Samples - PCBs (Surface Water)
QAPP Worksheet #28-2	June 2007	QC Samples - TSS (Surface Water)
QAPP Worksheet #28-3	June 2007	QC Samples - Phosphorus (Surface Water)
QAPP Worksheet #28-4	June 2007	QC Samples - PCBs (Soil/Sediment)
QAPP Worksheet #28-5	November 2008	QC Samples - PCBs (Free Product)
QAPP Worksheet #28-6	November 2008	QC Samples - TAL Metals (Groundwater)
QAPP Worksheet #28-7	November 2008	QC Samples - TAL Metals (Soil)
QAPP Worksheet #28-8	November 2008	QC Samples - Cyanide (Groundwater)
QAPP Worksheet #28-9	November 2008	QC Samples - Cyanide (Soil)
QAPP Worksheet #28-10	November 2008	QC Samples - Low-Level Mercury (Groundwater)
QAPP Worksheet #28-11	November 2008	QC Samples - SVOCs (Groundwater)
QAPP Worksheet #28-12	November 2008	QC Samples - SVOCs (Soil)
QAPP Worksheet #28-13	November 2008	QC Samples - VOCs (Groundwater)
QAPP Worksheet #28-14	November 2008	QC Samples - VOCs (Soil)
QAPP Worksheet #28-15	November 2008	QC Samples - DRO and GRO (Soil)
QAPP Worksheet #29	June 2007	Project Documents and Records
QAPP Worksheet #30	November 2008	Analytical Services
QAPP Worksheet #31-1	April 2008	Planned Project Assessments (Emergency Response Activities)
QAPP Worksheet #31-2	April 2008	Planned Project Assessments (Predesign Investigations for the Remedial Design For the 12th Street Landfill)
QAPP Worksheet #31-3	November 2008	Planned Project Assessments (Phase 2 of Addendum No. 1 [Test Pits] and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)
QAPP Worksheet #32-1	April 2008	Assessment Findings and Corrective Action Responses (Emergency Response Activities)
QAPP Worksheet #32-2	April 2008	Assessment Findings and Corrective Action Responses (Predesign Investigations for the Remedial Design For the 12th Street Landfill)
QAPP Worksheet #33	November 2008	QA Management Reports
QAPP Worksheet #34	April 2008	Verification (Step I) Process
QAPP Worksheet #35	April 2008	Validation (Step I) Process
QAPP Worksheet #36	November 2008	Validation (Steps IIa and IIb) Summary
QAPP Worksheet #37	April 2008	Usability Assessment

Acronyms and Abbreviations

µg/L	micrograms per liter
µg/kg	micrograms per kilogram
AA	atomic absorption
ARAR	Applicable, or Relevant and Appropriate Requirement
ASTM	American Society for Testing and Materials
BFB	p-bromofluorobenzene
BNA	base-neutral-acid extractable
CAS	Columbia Analytical Services
CCB	continuing calibration blank
CCC	calibration check compound
CCV	continuing calibration verification
CD	Consent Decree
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	Chain-of-Custody
COPC	constituent of potential concern
CVAA	cold vapor atomic absorption
DFTPP	decafluorotriphenylphosphine
DI	deionized
DO	dissolved oxygen
DQI	data quality indicators
DQO	Data Quality Objective
ECD	electron capture detector
EDD	electronic data deliverable
Eh	redox
FB	field blank
FS	Feasibility Study
FSP	Field Sampling Plan
GC/MS	gas chromatograph/mass spectrophotometer

HSP	Health and Safety Plan
ICB	initial calibration blank
ICP	inductively coupled plasma
ICPMS	inductively coupled plasma mass spectroscopy
ICS	interface check samples
ICV	initial calibration verification
IDW	investigation-derived waste
kg	kilogram
KRSG	Kalamazoo River Study Group
L	liter
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LRA	linear range analysis
MDEQ	Michigan Department of Environmental Quality
MDL	Method Detection Limit
MS	matrix spike
MS/MSD	matrix spike/matrix spike duplicate
MSD	matrix spike duplicate
NCP	National Contingency Plan
NIST	National Institute of Standards and Technology
NTU	nephelometric turbidity unit
OSC	On-Scene Coordinator
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
pH	negative logarithm (base 10) of hydrogen ion activity
PID	photoionization detector
PQO	Project Quality Objective
POTW	publicly-owned treatment works
PM	Project Manager
QA	quality assurance
QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
QC	quality control
QL	Quantitation Limit

RASs	routine analytical service
RD/RA	Remedial Design/Remedial Action
RF	response factor
RI	Remedial Investigation
RI/FS	Remedial Investigation/Feasibility Study
ROD	Record of Decision
RPD	relative percent difference
RL	reporting limit
RPM	Remedial Project Manager
RSD	relative standard deviation
RT	retention time
SAP	Sampling and Analysis Plan
SAS	special analytical services
SOP	standard operating procedure
SOW	Statement of Work
SPCC	system performance check compound
SRI/FS	Supplemental Remedial Investigation/Feasibility Study
SRM	standard reference material
SW846	Test Methods for Evaluating Solid Waste, 1996
TAL	Target Analyte List
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TCRA	Time-Critical Removal Action
TEMP	temperature
TB	trip blank
TSS	Total suspended solids
U.S. EPA	United States Environmental Protection Agency
VOC	volatile organic compound
WATS	Weyerhaeuser Analytical Testing Services

Section 1
Introduction

On February 22, 2005, Weyerhaeuser Company (Weyerhaeuser) entered into a Consent Decree (CD) with the United States Environmental Protection Agency (U.S. EPA) for the Design and Implementation of Certain Response Activities at the 12th Street Landfill site (Operable Unit No. 4) and the Plainwell Mill site (Operable Unit No. 7). Both sites are part of the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund site (Figure 1-1), which is located in southwestern Michigan. A Statement of Work (SOW) for the Remedial Design/Remedial Action (RD/RA) at the 12th Street Landfill site was attached to the CD. A SOW for the Remedial Investigation/Feasibility Study (RI/FS) at the Plainwell Mill site was subsequently issued by the U.S. EPA, with an effective date of August 17, 2006.

In late February 2007, the U.S. EPA authorized a Time-Critical Removal Action (TCRA) to remove polychlorinated biphenyl (PCB)–contaminated sediment in the former Plainwell Impoundment (a section of Operable Unit #5 of the Allied Paper/Portage Creek/Kalamazoo River Superfund Site). This work was authorized through an administrative settlement agreement and Order on Consent for Removal Action (V-W-07-C-8-63). As part of the TCRA, the earthen section of the Plainwell Dam was removed and the Kalamazoo River was rerouted through the former powerhouse discharge channel. The 12th Street Landfill abuts the river and is located directly downstream of the earthen section of the Plainwell Dam on the western side of the former powerhouse discharge channel. The Plainwell Mill also abuts the Kalamazoo River and PCB-containing materials have been documented along the riverbank at the Mill. The change in the Kalamazoo River channel will result in an increased river gradient and higher velocities upstream and along the rerouted channel (USGS, 2004 and USDA, 2004) and are expected to flush residuals in the former powerhouse discharge channel downstream and erode bank material in the area of the Mill. Thus, the scope of the TCRA activities included actions or occurrences that threatened releases of waste material (as defined in the CD) from both the 12th Street Landfill and the Plainwell Mill properties. Since any such release may present an immediate threat to public health or welfare of the environment, Weyerhaeuser was authorized to conduct several emergency response actions in conjunction with completing the required work under the CD.

The U.S. EPA requires that all environmental monitoring and measurement efforts mandated or supported by the U.S. EPA participate in a centrally managed quality assurance program. Any party generating data under this program has the responsibility to implement minimum procedures to ensure that the precision, accuracy, completeness, and representativeness of the data are known and documented. To ensure that this responsibility is met uniformly, a written Quality Assurance Project Plan (QAPP) must be prepared for each project.

This Multi-Area QAPP presents the objectives, organization, functional activities, and specific quality assurance (QA) and quality control (QC) activities associated with implementing the projects that are described in Section 2. This QAPP also describes the specific protocols that will be followed for sampling, sample handling and storage, chain-of-custody, and laboratory analysis. This Multi-Area QAPP will be modified in the future as other sampling programs are identified or defined (*e.g.*, additional worksheets, such as Problem Definition-DQOs [Worksheet No. 10], will be inserted to describe the project sampling objectives and sampling program).

1.1 Development of the Multi-Area QAPP

This QAPP has been prepared in accordance with the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP), Manual V1, March 2005 (U.S. EPA, 2005a), and the Uniform Federal Policy for Quality Assurance Project Plans, Part 2B, Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities V1, March 2005 (U.S. EPA, 2005b).

All QA/QC procedures performed under this QAPP will be in accordance with applicable professional technical standards, U.S. EPA requirements, other pertinent government regulations and guidelines, and the specific project goals and requirements.

1.2 Project Setting

The Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund site is located in southwestern Michigan. The site includes five disposal areas, five paper mill properties, an approximately 80-mile stretch of the Kalamazoo River from Morrow Dam to Lake Michigan, and a 3-mile stretch of Portage Creek. The primary site contaminant is polychlorinated biphenyls (PCBs), a hazardous substance and probable human carcinogen. PCBs were introduced to Portage Creek and the Kalamazoo River through past discharges and disposal of PCB-contaminated residuals.

The 12th Street Landfill and Plainwell Mill sites are located in Allegan County, Michigan (Figure 1-2). The 12th Street Landfill is located in Otsego Township (Section 24, Township 1N, Range 11W), and the Plainwell Mill is located in the City of Plainwell (Section 30, Township 1N, Range 11W). Both sites are located adjacent to the Kalamazoo River, with the 12th Street Landfill located approximately 1½ miles northwest and downstream of the Plainwell Mill site. The 12th Street Landfill site encompasses approximately 6.5 acres and is situated on roughly a 24-acre property that is bordered to the east by woodlands and a former hydroelectric powerhouse discharge channel on the Kalamazoo River, to the north and west by wetlands, to the south and southwest by a asphalt plant mining operation, and to the south and southeast by industrially developed lands and the Plainwell Dam (which is scheduled to be removed as part of the U.S. EPA-approved TCRA in 2007-2008). The Plainwell Mill site encompasses approximately 36 acres and is bordered by the Kalamazoo River to the north, the Plainwell central

business district to the east, residential properties to the south, and commercial properties and the City of Plainwell wastewater treatment plant to the west.

Plainwell, Inc., is the current owner of the 12th Street Landfill property, although Plainwell, Inc., is a bankrupt entity with no ongoing business operations. Weyerhaeuser is currently in negotiations with Plainwell, Inc., to take ownership of the 12th Street Landfill property. The mill property has been vacant since the former Simpson Plainwell Paper Company filed for bankruptcy in 2000. The City of Plainwell is the current owner of the Plainwell Mill property, having purchased the property out of the Plainwell Mill bankruptcy in 2006. Weyerhaeuser owned and operated the mill and the landfill for approximately a 9-year period, between 1961 and 1970. During that time, dewatered sludge from wastewater treatment operations was excavated from lagoons on the mill property and transported for disposal at the 12th Street Landfill site.

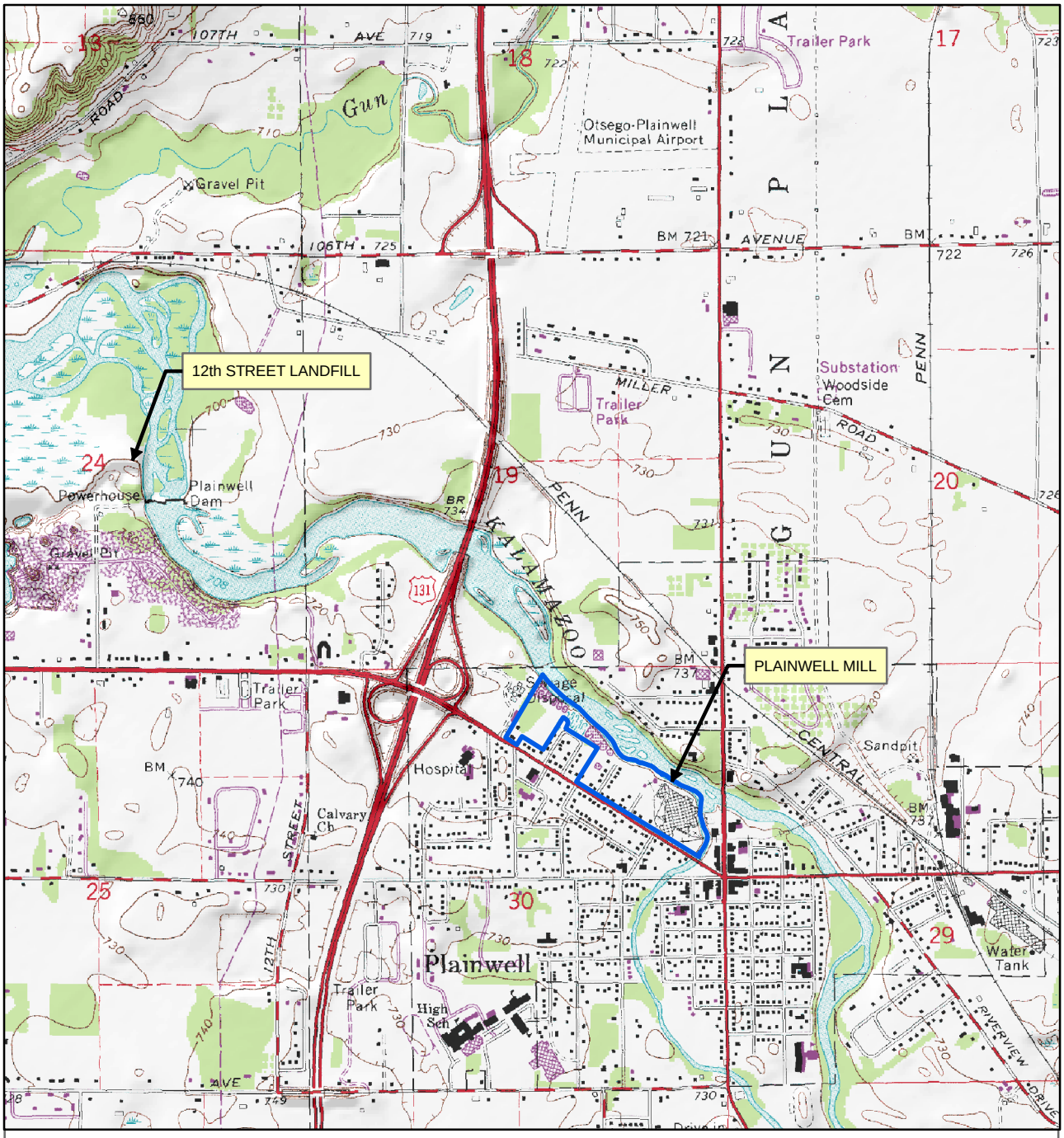
1.3 Applicability of the Multi-Area QAPP

This Multi-Area QAPP is applicable for work to be performed by Weyerhaeuser and its representatives within the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund site. The work activities will include tasks conducted by Weyerhaeuser under its 2005 CD with the U.S. EPA, as may be amended or modified, at the following sites:

- 12th Street Landfill (Operable Unit No. 4)
- Plainwell Mill (Operable Unit No. 7)

Additional areas may be addressed in the future through modifications of this Multi-Area QAPP. This QAPP may be updated to address additional emergency activities and/or to integrate applicable workplan activities as required under the CD.

In September 2007, this QAPP was amended to include the removal of residuals and sediment along certain sections of the banks along the Plainwell Mill property. As defined in the 2005 CD, the limits of the Plainwell Mill Operable Unit (OU-7) extend to the top of the riverbank. Consequently, the banks of the mill property are part of the Kalamazoo River Operable Unit (OU-5).



LEGEND

PROPERTY BOUNDARIES

0 1,000 2,000 4,000 FEET

1 INCH EQUALS 2,000 FEET

1:24,000

N

PROJECT LOCATION

BASE MAP FROM USGS 7.5 MINUTE QUADRANGLE, OSTEGO, 1967, REVISED 1973.

RMT

744 Heartland Trail
Madison, WI 53717 - 1934

P.O. Box 8923
Madison, WI 53708 - 8923
Phone: 608-831-4444
Fax: 608-831-3021

SITE LOCATION MAP

WEYERHAEUSER COMPANY
MULTI-AREA QAPP
PLAINWELL, MICHIGAN

DRAWN BY:	PAPEZ J
APPROVED BY:	LEH
PROJ. NO.:	00-005117.05
FILE NO.:	51170501.mxd
DATE:	APRIL 2008

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FIGURE 1-1

RMT
744 Heartland Trail
Madison, WI 53717 - 1934
P.O. Box 8923
Madison, WI 53708 - 8923
Phone: 608-831-4444
Fax: 608-831-3021

FIGURE 1-2
ALLIED PAPER, INC.
PORTAGE CREEK
KALAMAZOO RIVER SUPERFUND SITE

WEYERHAEUSER COMPANY
MULTI-AREA QAPP
PLAINWELL, MICHIGAN

DRAWN BY:	PAPEZ J
APPROVED BY:	LEH
PROJ. NO.:	00-005117.05
FILE NO.:	51170502.mxd
DATE:	APRIL 2008

1 INCH EQUALS ~4 MILES

SITE LOCATION

86°15'0"W 86°0'0"W 85°45'0"W 85°30'0"W 85°15'0"W

42°30'0"N 42°15'0"N

MICHIGAN

LAKE

Saugatuck

Fennville

Kalamazoo River

OU5: KALAMAZOO RIVER

Allegan County

Barry County

Allegan Dam

Allegan

Allegan City Dam

Ostego City Dam

Plainwell Dam

Trowbridge Dam

Otsego

OU7: PLAINWELL MILL

Ostego Dam

Plainwell

OU4: 12th STREET LANDFILL

South Haven

South Haven

Van Buren County

Gobles

Kalamazoo County

OU3: KING HIGHWAY LANDFILL

Fort James Paper Mill

Parchment

Georgia Pacific Mill

Springfield

Morrow Dam

Portage Creek

Battle Creek

Kalamazoo

Galesburg

OU1:
ALLIED PAPER
LANDFILL

OU2:
WILLOW BLVD /
A-SITE LANDFILL

Allied Paper Mill

Bangor

Hartford

Coloma

Watervliet

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Section 2
Scope of Activities

The subsections below provide a summary of the scope for each project conducted under this Multi-Area QAPP to date, as well as new projects being added in this version. These projects include the following:

1. Emergency response activities in the former powerhouse discharge channel at the 12th Street Landfill (as described in Revision 00, June 2007, and Revision 01, August 2007)
2. Emergency Response activities on portions of the riverbank along the Plainwell Mill (as described in Revision 02, Addendum 01, September 2007)
3. Predesign investigations for the remedial design for the 12th Street Landfill (added in this version of the Multi-Area QAPP [i.e., Revision 02, Addendum 02, February 2008])

The summaries of projects included in previous versions of this document have not been revised or deleted. As new projects are initiated, new information will be added to this Multi-Area QAPP to cover the new projects, and a new addendum number will be assigned to the document (*e.g.,* Addendum 02 will be updated to Addendum 03, and so on). If information included in a previous version of this document is changed, a new revision number will be assigned (*e.g.,* Revision 02 will be updated to Revision 03, and so on).

2.1 Emergency Response Activities at the 12th Street Landfill (Operable Unit No. 4)

Weyerhaeuser has not yet received a formal notification from the U.S. EPA to proceed with the 12th Street Landfill Remedial Design/Remedial Action (RD/RA) work specified in the CD. The full scope of the Remedial Action (RA) for the 12th Street Landfill generally includes the excavation of wastewater residuals present outside the footprint of the landfill (on adjacent properties and in the former powerhouse discharge channel); the relocation of the excavated material back into the landfill, the construction of a landfill cover and erosion protection measures, and various environmental monitoring activities. Among the paper residuals to be excavated are those present in the former powerhouse discharge channel (a backwater of the Kalamazoo River), to the extent that these residuals are visibly contiguous with the landfill.

The TCRA in the Former Plainwell Impoundment includes the removal of the earthen section of the Plainwell Dam. The TCRA was approved by the U.S. EPA in February 2007, and the specified work will be implemented by the Kalamazoo River Study Group (KRSG) in 2007 and 2008 (Arcadis BBL, 2007a). Consequently, Weyerhaeuser needs to conduct certain activities at the 12th Street Landfill site under Section XVII, paragraph 67 (Emergency Response), of Weyerhaeuser's CD with the U.S. EPA in order to accommodate the TCRA and to prevent erosion of the banks of the landfill. These activities include

removing visible residuals in the former powerhouse discharge channel, cutting back the existing landfill slope along the river, and installing erosion protection on the eastern bank of the 12th Street Landfill consistent with the requirements of the Record of Decision (ROD).

2.1.1 Former Powerhouse Discharge Channel

The 12th Street Landfill is situated directly downstream of the Plainwell Dam, on the same side of the river as the earthen section of the dam that will be removed as part of the TCRA. After the earthen section of the Plainwell Dam is removed, the former powerhouse discharge channel (currently a backwater) will become the main channel of the Kalamazoo River. This will create a larger gradient on the river with resultant higher flow velocities and greater shear stresses (*i.e.*, erosion potential) in the former powerhouse discharge channel and on various bank areas upstream of the current Plainwell Dam. The change will also alter flood conditions near the landfill. Without action, the channel rerouting will resuspend and redistribute paper residuals that are present in the discharge channel, including residuals that may be contiguous with the 12th Street Landfill. The modified flow is also modeled to increase shear stresses along the riverbank adjacent to the 12th Street Landfill, resulting in possible destabilization of the banks and increased erosion. To prevent this from occurring, Weyerhaeuser plans to perform the following activities:

- Conduct bathymetric and visual sediment assessments to determine the visual characteristics of the sediment and the depth of water in the former powerhouse discharge channel (RMT, 2007a). Use this information, in conjunction with previously available information (G&M, 1994; U.S. EPA, 2004), to estimate the extent of visual wastewater residuals that are contiguous with the 12th Street Landfill, present in the channel sediment, and subject to mobilization once the Kalamazoo River is rerouted.
- Excavate visible wastewater residuals that are contiguous with the 12th Street Landfill and present in the channel sediment. Dewater the sediment, and treat and sample water from the dewatering process before discharge back to the Kalamazoo River. Dispose the excavated sediment in the 12th Street Landfill.
- Collect post-excavation confirmation samples in the former powerhouse discharge channel to confirm that wastewater residuals that are visibly contiguous with the landfill have been removed, and that PCB concentrations in the excavated area are below 4 mg/kg.

The locations and concentration ranges of data collected previously at the site, as well as an indication of whether residuals were observed, are illustrated on Figure 2-1.

2.1.2 Eastern Bank of the Landfill

As stated in Subsection 2.1.1, after the earthen section of the Plainwell Dam is removed, the former powerhouse discharge channel will become the main channel of the Kalamazoo River. This will create higher velocities and greater shear stresses (*i.e.,* erosion potential), as well as altered flood conditions along the eastern bank of the 12th Street Landfill. Without action, the

channel rerouting could erode mixed fill/paper residuals at the toe of the eastern landfill bank and may lead to instability of the bank. Moreover, certain components of the ROD, such as cutting back the sideslope of the landfill along the river and creating space for an access road along the river, are more effectively and efficiently implemented in conjunction with the work in the former discharge channel. Consequently, Weyerhaeuser plans to perform the following activities that are required by the ROD for the 12th Street Landfill as part of an Emergency Response Action:

- Cut back the existing slope of the 12th Street Landfill along the river to create a more stable angle and to provide a buffer zone to ensure that, once the final cover system is installed, there is no direct contact between the paper residuals in the landfill and the Kalamazoo River/former powerhouse discharge channel, and to provide space for an access road along the riverfront. The excavated mixed fill/paper residuals from the eastern bank will be disposed in a designated area on top of the 12th Street Landfill.
- Install erosion protection on the newly graded eastern bank of the landfill to provide protection from a 500-year flood event.

Geotechnical borings have been installed in preparation for this work (RMT, 2007b). Consistent with the requirements of the ROD, no environmental samples are planned in connection with the above work at the 12th Street Landfill.

The final cover over the landfill, along with the other components of the landfill remedy, will be designed and installed in a manner and time frame consistent with the CD.

2.2 Emergency Response Activities at the Former Plainwell Mill Banks (Operable Unit No. 5)

Weyerhaeuser has been authorized by the U.S. EPA to conduct Emergency Actions at two separate locations adjacent to the Kalamazoo River under Paragraph 67 of their 2005 Consent Decree. As part of the Emergency Action for the former powerhouse channel at the 12th Street Landfill (Operable Unit 04), Multi-Area QAPP and Field Sampling Plan (FSP) were submitted and approved (approval letter from Michael Berkoff, dated July 24, 2007). On June 29, 2007, the U.S. EPA also authorized Weyerhaeuser to take actions to prevent, abate, or minimize a release or potential release of hazardous substances from the former Plainwell Mill banks. The former Plainwell Mill banks are considered part of the Kalamazoo River operable unit (Operable Unit 05).

The following objectives have been developed for the Emergency Action along the former Plainwell Mill banks:

- Remove or contain visible paper residuals and address previously identified areas of reported polychlorinated biphenyls (PCBs) concentrations of greater than 50 mg/kg in soils/sediments along the former Plainwell Mill banks to a target concentration of 4 mg/kg.
- Reconstruct bank, as needed, to minimize future release of PCBs.

- Reconfigure bank to limit upland cutbacks into former Plainwell Mill property and place erosion controls to provide comparable stability to pre-excavation conditions.

To achieve the excavation objectives stated above, physical and analytical data from previous investigations performed on sediments, bank soils, and floodplain soils were reviewed to identify and delineate specific areas of sediment and soil to be targeted as part of this Emergency Action. The design approach to the Emergency Action is presented in a draft final Plainwell Mill Banks Emergency Action Design Report (RMT, 2007c). Site preparation work is scheduled to begin late September 2007 with construction to follow in early October. The 2,600 linear feet of bank length has been divided into four zones based upon physical conditions and estimated quantity of visual residuals targeted for excavation. These construction zones will facilitate construction by providing an opportunity to refine the construction approach and schedule between each zone.

2.3 Predesign Investigations for the Remedial Design for the 12th Street Landfill (Operable Unit No. 4)

The 12th Street Landfill, composed primarily of the 6.5-acre closed landfill and the four areas outside the landfill where PCB-contaminated residual material has been observed, is Operable Unit No. 4 (OU-4) of the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site (Kalamazoo River Superfund Site). The landfill is situated with 17 acres of wetlands on an approximately 24-acre parcel. Additional portions of OU-4 that are located outside the landfill property include the woodland area owned by the State of Michigan (State) under the management of the Michigan Department of Natural Resources (MDNR), the asphalt plant operation adjacent to the landfill, and the former powerhouse discharge channel. Contamination in the former powerhouse discharge channel was addressed as part of Emergency Actions implemented in 2007.

Paper residuals from the former Plainwell Mill (mill), located in Plainwell, Michigan, were disposed in the 12th Street Landfill during the period from approximately 1955 to 1983. The landfill reportedly also accepted solid waste from the mill during part of its period of active operation. A number of investigations have been performed at the site. The investigations have confirmed the nature of the material in the landfill and have shown that paper residuals are present in certain areas outside of the landfill (*i.e.,* in the wetlands to the north/northwest, the asphalt plant property, and the State property). Some of the residuals/native soil beyond the toe of the landfill (*i.e.,* outside the footprint of the landfill) may contain PCBs at concentrations exceeding State of Michigan or Kalamazoo River Superfund Site-specific ecological risk-based criteria.

A ROD for OU-4 was issued on September 28, 2001. The major components of the selected remedy include the following: "(1) the excavation and relocation into the landfill of contaminated residuals currently in the woodland, wetlands, and adjacent property, and the residuals in the former powerhouse discharge channel that are contiguous with the eastern side of the landfill, followed by the construction of a

containment system; (2) the excavation and relocation into the landfill of the east side of the landfill along the former powerhouse discharge channel to create a buffer zone sufficient to insure no hydraulic connection between the wastes within the landfill containment system and the Kalamazoo River or the former powerhouse discharge channel; (3) the restoration of areas excavated, cleared and grubbed, or otherwise affected by the remedial action; (4) the construction of a side wall containment system around the outside of the landfill; (5) the construction of a cover (cap) over the landfill; (6) the installation of an appropriate groundwater monitoring network and the performance of long-term groundwater monitoring; (7) the performance of short-term surface water monitoring; (8) the placement of deed restrictions; (9) the construction of a fence, permanent markers, and warning signs; (10) the investigation of the need for a leachate collection system and, if deemed necessary, the construction of a leachate collection system; and (11) the implementation of long-term maintenance and post-closure care."

In January 2005, Weyerhaeuser negotiated a CD with the U.S. EPA (Civil Action No. 1:05-CV0003) for the design and implementation of certain response actions at Operable Unit #4 and the Plainwell, Inc., Mill. These actions include the RD/RA activities for the 12th Street Landfill. This revision of the Multi-Area QAPP has been prepared in fulfillment of the requirements for an RD Workplan for the 12th Street Landfill that are contained in the CD and the SOW.

The RD Workplan for the 12th Street Landfill includes a number of predesign investigations, some of which will involve field data collection and visual observations. Field data collection activities will include visual information obtained by the advancement of test pits, visual information obtained by the advancement of Geoprobe® borings, and gas concentration measurements (methane, carbon dioxide, and oxygen) in existing monitoring wells and in the Geoprobe® borings advanced as part of the predesign investigation.

The field investigations will involve: 1) a determination of the extent of visible paper residuals beyond the landfill footprint, 2) the collection of data to support the grading design for the final landfill cover, and 3) the collection of data for use in the design of a landfill gas venting system. Sufficient information is available from previous investigations to evaluate the potential need for a leachate collection system. No additional field information is needed to support this objective.

2.3.1 Extent of Paper Residuals Beyond Landfill Footprint

The objectives of this investigation include the following:

Wetland

- To confirm the approximate areal extent of visible paper residuals beyond the toe of the landfill as delineated based on the results from previous investigations.
- To assess the degree of difficulty in distinguishing the visible paper residuals from the native soil.

- To evaluate potential constructibility issues associated with working in the wetland north of the landfill.

Asphalt Plant/State Properties

- To delineate the areal extent, and to better estimate the depth, of visible paper residuals on the asphalt plant property to the southwest and on the State property to the southeast, in order to support discussions with owners of these adjacent properties concerning access for conducting the remedial actions on the properties required by the ROD.

The general scope of the predesign investigation includes the following activities:

Wetland

- Excavate approximately three test pits at the locations depicted on Figure 2-2. The test pits are anticipated to be approximately 10 to 15 feet long (perpendicular to the edge of the landfill) and approximately 2 to 4 feet wide. The test pits will be excavated to a maximum depth of 3 feet if no paper residuals are visually apparent, or to the bottom of visually-identifiable residuals.

Asphalt Plant/State Properties

- Excavate approximately three test pits on the asphalt plant property, and approximately four test pits on the State property at the locations depicted on Figure 2-2. The test pits are anticipated to be approximately 10 to 15 feet long (perpendicular to the edge of the landfill) and approximately 2 to 4 feet wide. The test pits will be excavated to a maximum depth of 3 feet if no paper residuals are visually apparent, or to the bottom of visually-identifiable residuals.

  In the event that in-field conditions limit the use of test pit excavating equipment (*e.g.,* a backhoe), other tools, such as hand augers or shovels, may be used instead. In such instances, the U.S. EPA project manager, or designated alternate, will be contacted to discuss the situation and to agree on a course of action. In addition, Weyerhaeuser will conduct a kick-off meeting with the U.S. EPA and its field representative(s) at the outset of the predesign field investigation. During this meeting, the schedule for conducting the predesign field activities and the process for obtaining U.S. EPA approval of field modifications will be reviewed.

2.3.2 Grading Design

The objective of this investigation is the following:

- To better estimate the depth of the paper residuals along the property boundaries with 12th Street, the asphalt plant to the southwest, and with the State property to the southeast, in order to reduce uncertainties in designing the final landfill grades.

The general scope of this investigation is the following:

- Advance approximately nine Geoprobe® borings into the 12th Street Landfill at locations where fill material is believed to extend beyond the property boundaries to the southwest and to the southeast (see locations on Figure 2-3). The borings will be advanced approximately 5 feet into the native soil underlying the fill, or to refusal.
- Advance a minimum of two soil borings (RDB-10 and RDB-11) near the southern end of the landfill, as shown on Figure 2-3, to confirm the thickness of the fill in this area. Advance the borings approximately 5 feet into the native soil underlying the fill or to refusal. The locations of these borings may be adjusted in the field as necessary to avoid underground or aboveground utility lines. Additional borings may be installed to the north of the initial borings as may be deemed useful by Weyerhaeuser, in consultation with oversight agencies as needed, for purposes of designing the landfill cover (*e.g.,* if fill material is not encountered at a location where existing data indicate that fill is present).

2.3.3 Landfill Gas Management

The objective of this investigation is the following:

- To collect readily accessible information about the subsurface landfill gas conditions at the 12th Street Landfill that may be useful in designing a passive gas venting system for the 12th Street Landfill.

The general scope of this field investigation is the following:

- Measure the concentrations of methane, carbon dioxide, and oxygen in the existing groundwater monitoring wells at the 12th Street Landfill that are screened in the vadose zone (MW-6A, MW-7A, and MW-8A), and in the Geoprobe® boreholes used to estimate the depth of the paper residuals along the property boundaries (see locations on Figure 2-3). Pressures that may have developed within the groundwater monitoring wells caused by excess landfill gas (if present) will also be measured. A passive gas venting system can be designed without the above information. If these data cannot be readily obtained, additional efforts will not be employed to collect the information.

2.4 Phase 2 of Addendum No. 1 Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment (Operable Unit No. 7)

2.4.1 Phase 2 of Addendum No. 1

In a letter to Weyerhaeuser dated February 19, 2008, the U.S. EPA requested additional work along the former Plainwell Mill banks in response to discovery of elevated concentrations of PCBs and oily soil encountered along the Plainwell Mill banks during the separate Plainwell Mill Banks Emergency Response activities. On July 15, 2008, a Draft Phase 2 Addendum No. 1 to the Plainwell Mill Remedial Investigation/Feasibility Study (RI/FS) Work Plan was prepared and provided to U.S. EPA for review. The U.S. EPA provided technical review comments on the Draft Phase 2 Addendum No. 1 in a letter dated July 31, 2008. Subsequently, Weyerhaeuser provided response to comments and a redlined version of the Work Plan Addendum in a letter

dated August 8, 2008. The Phase 2 of Addendum No. 1 Work Plan has been designed to meet the following objectives:

1. Identify Source: Identify, as much as possible, a likely cause of the oily sheen observed near the former transformer pad, with focus on the possible presence of NAPL.
2. Determine Relationships: Determine if there is a relationship between the oily material near the transformer pad and the elevated PCB concentrations found at PM-SD-041, PEX-1, and PEX-2 (Figure 2).
3. Assess NAPL: Determine whether NAPL, if present, is entering the Kalamazoo River.
4. Define Nature of Contamination: Determine if the oily material present in soils and fill near the transformer pad and at locations PM-SD-041, PEX-1, and PEX-2 contain petroleum products and/or PCBs.
5. Develop Next Steps: Determine immediate actions needed and how the next phase of investigation should be integrated with other RI activities.

Due to the nature of the investigate areas likely to contain fill material and or construction debris, test pits have been selected as the best method for successful site characterization. Test pits will be advanced to either native soils or the water table using an excavator taking care to minimize site disturbances to the extent practical. Additional details and the rationale for the proposed test pit investigation are presented in the Phase 2 of Addendum No. 1 Work Plan and the associated QAPP Worksheet #10-4.

2.4.2 Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment

The Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment includes a collection of shallow groundwater samples to assess the possible presence of floating product and to quantify the concentrations of constituents in shallow groundwater that may have been contributed by suspected or known contamination present on site. The Plainwell Mill is located at 200 Allegan Street in Plainwell, Michigan. The limited existing groundwater data, the generally permeable subsurface sands present along the Kalamazoo River, the site history, and the size and configuration of the Plainwell Mill property make a phased groundwater investigation especially useful for focusing future data collection activities needed to meet the overall objectives of the required RI/FS.

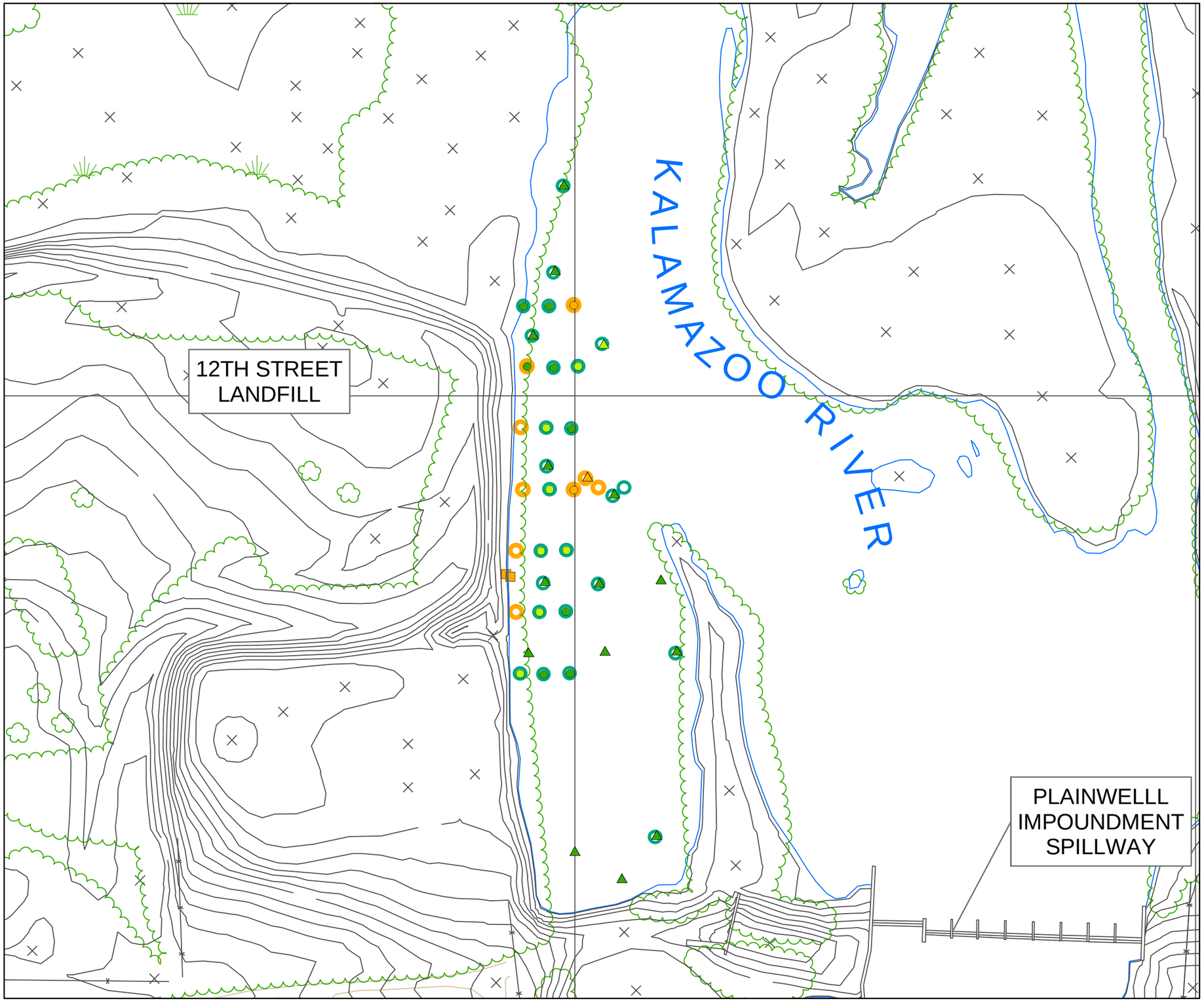
The specific objectives for the Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment are:

- To determine if free product is present on the groundwater table at the monitoring well locations.
- To evaluate shallow groundwater quality across the Plainwell Mill property.
- To assess shallow site-specific hydro-geologic characteristics that will be used to refine the current site conceptual model.

- To determine the potential usability of past site data in the determination of the nature and extent of contamination present on the Plainwell Mill.
- To identify data gaps that need to be addressed during Phase II investigations of soil and groundwater.

Groundwater sampled from each location will be analyzed for volatile organic compounds (VOCs), polycyclic aromatic hydrocarbons (PAHs), RCRA metals, and PCBs. As a frame of reference, concentrations of measured constituents will be compared to applicable Michigan standards.

The results of this phase of the RI will also be used to help assess the potential usefulness of groundwater results from previous environmental assessments conducted on the site, better define site conditions, and then allow identification of data gaps. The identified data gaps will then be addressed during the Phase 2 soil and groundwater sampling activities expected for this site. Additional details and the rationale for the phased groundwater assessment are presented in the Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment Work Plan and the associated QAPP Worksheet #10-4.



LEGEND

BBL Data (2001) - Total PCBs mg/kg (USEPA, 2004)

- 0 - 1
- 1 - 4
- 4 - 50
- 50+

USEPA Data 2004 - Total PCBs (mg/kg)

- 0 - 1
- 1 - 4
- 4- 50
- 50+

Geraghty and Miller (1994) - Total PCBs (mg/kg)

- PCB Concentration 17 & 29 ppm

Powerhouse Channel Residuals

- No Visible Residuals
- Visible Residuals

NOTE

1. Concentrations presented are maximum at each sample location.

N

0 40 80 160 FEET

1:960

1 INCH EQUALS 80 FEET

PROJECT:	WEYERHAEUSER COMPANY MULTI-AREA QAPP PLAINWELL, MICHIGAN		
SHEET TITLE:	HISTORICAL SAMPLING DATA FOR THE FORMER POWERHOUSE DISCHARGE CHANNEL		
DRAWN BY: WEBERN	SCALE: AS NOTED	PROJ. NO.: 00-05117.03	
CHECKED BY: MK		FILE NO.: 51170309.mxd	
APPROVED BY: CJ	DATE PRINTED: 4/24/2008	**FIGURE 2-1**	
DATE: APRIL 2008			

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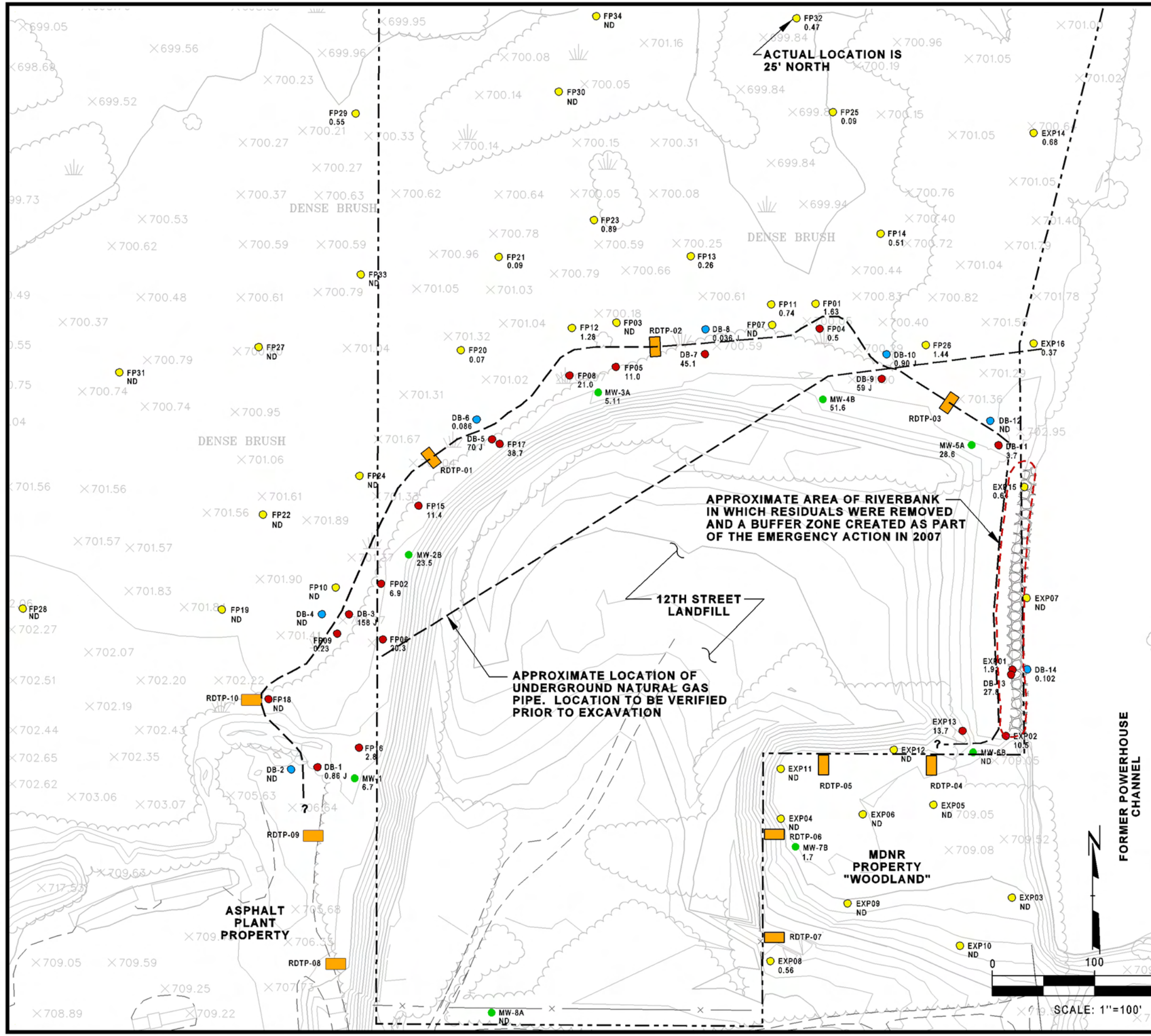

LEGEND

- RDTP-01 — PROPOSED TEST PIT
- BORING LOCATION AT WHICH VISIBLE PAPER RESIDUALS WERE NOT PRESENT IN UPPER 24 INCHES
- BORING LOCATION AT WHICH VISIBLE PAPER RESIDUALS WERE DOCUMENTED IN BORING LOGS
- BORING LOCATION AT WHICH VISIBLE PAPER RESIDUALS WERE PRESUMED TO NOT HAVE BEEN IDENTIFIED (GIVEN THE OBJECTIVE OF THE INVESTIGATION), BUT WERE NOT DOCUMENTED (BORING LOGS ARE NOT AVAILABLE)
- MONITORING WELL
- APPROXIMATE PROPERTY BOUNDARY
- APPROXIMATE LIMITS OF PAPER RESIDUALS USING VISUAL CRITERIA

NOTES

1. RESULTS FOR THE "EXP" AND "FP" SAMPLING SERIES PROVIDED BY THE USEPA. THE SAMPLING WAS CONDUCTED BY THE USEPA IN SEPTEMBER AND OCTOBER 2003.
2. RESULTS FOR THE "DB-" SAMPLING SERIES ARE FROM THE ALLIED PAPER, INC./PORTAGE CREEK / KALAMAZOO RIVER SUPERFUND SITE REMEDIAL INVESTIGATION, TECHNICAL MEMORANDUM 8, GERAGHTY AND MILLER, INC. MAY 31, 1994.
3. THE APPROXIMATE LIMITS OF VISIBLE RESIDUALS CRITERIA WAS DERIVED FROM THE ALLIED PAPER, INC./PORTAGE CREEK / KALAMAZOO RIVER SUPERFUND SITE REMEDIAL INVESTIGATION, TECHNICAL MEMORANDUM 8, (GERAGHTY AND MILLER, INC. MAY 31, 1994.) AND REVISED BASED ON THE U.S.EPA'S 2003 PRECONSTRUCTION INVESTIGATION AND ON THE REMOVAL ACTIONS PERFORMED ALONG THE RIVERBANK IN 2007 AS PART OF THE EMERGENCY ACTION.
4. THE CONCENTRATIONS SHOW THE MAXIMUM CONCENTRATION (MG/KG) OF TOTAL PCBS IN EACH DEPTH INTERVAL AT EACH SAMPLE LOCATION.
5. PROPERTY BOUNDARY BASED ON LEGAL DESCRIPTION PROVIDED BY THE USEPA IN A FAX DATED MARCH 30, 2004. COORDINATES FOR S1/4 CORNER AND N 1/4 CORNER OF SECTION 24 AND NORTH BEARING USED TO PLOT PROPERTY LINE WERE PROVIDED BY HOLLAND ENGINEERING AND ARE BASED ON MICHIGAN STATE PLANE, SOUTH ZONE, NAD 83, US SURVEY FOOT COORDINATES.
6. CONCENTRATIONS QUALIFIED WITH A "J" FLAG ARE ESTIMATED. THE TOTAL CONCENTRATIONS ARE UNCERTAIN.

PROJECT: **12TH STREET LANDFILL MULTI-AREA QAPP - REVISION 2 OTSEGO TOWNSHIP, MICHIGAN**

SHEET TITLE: **PROPOSED WETLAND, WOODLAND, AND ASPHALT PLANT AREA PREDESIGN INVESTIGATIONS**

DRAWN BY: stormerl	SCALE: 1"=100'	PROJ. NO.	5117.02 \ Qapp
CHECKED BY: ECW		FILE NO.	PRO INVEST.PLT
APPROVED BY: LEH	DATE PRINTED:	**FIGURE**	**2-2**
DATE: APRIL 2008			

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Phone: 608.831.4444
Fax: 608.831.3334

Reference Files
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Ref. File 2 - J:\05117\02\Qapp\bmrmt.dgn (2) bmr
Ref. File 3 - J:\05117\02\Qapp\proposed.dgn (3) pro
Ref. File 4 - J:\05117\02\Qapp\11x17bdr.dgn (4) border
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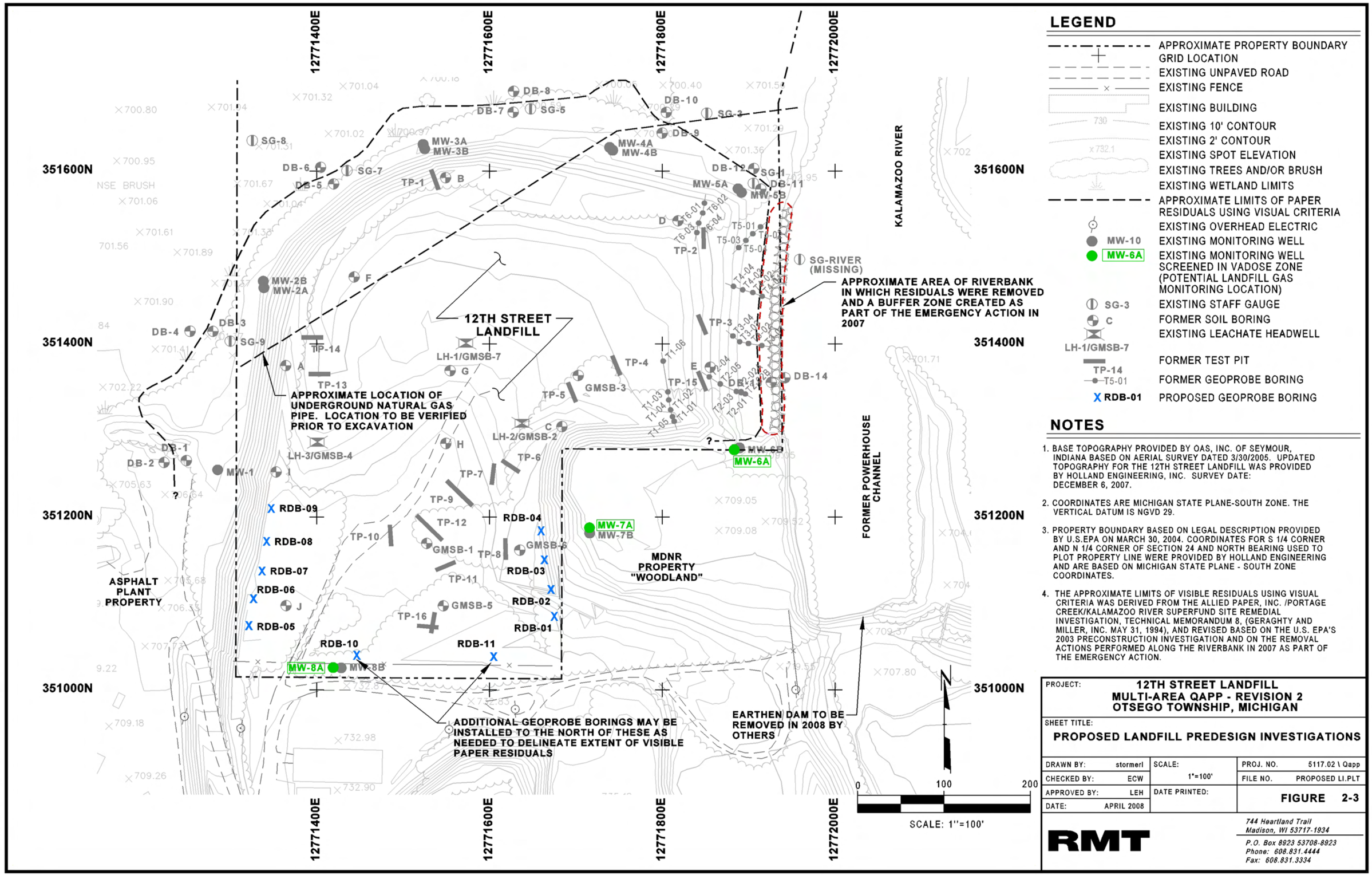
Levels On - 1-63



LEGEND

- APPROXIMATE PROPERTY BOUNDARY
- GRID LOCATION
- EXISTING UNPAVED ROAD
- EXISTING FENCE
- EXISTING BUILDING
- EXISTING 10' CONTOUR
- EXISTING 2' CONTOUR
- EXISTING SPOT ELEVATION
- EXISTING TREES AND/OR BRUSH
- EXISTING WETLAND LIMITS
- APPROXIMATE LIMITS OF PAPER RESIDUALS USING VISUAL CRITERIA
- EXISTING OVERHEAD ELECTRIC
- MW-10 EXISTING MONITORING WELL
- MW-6A EXISTING MONITORING WELL SCREENED IN VADOSE ZONE (POTENTIAL LANDFILL GAS MONITORING LOCATION)
- SG-3 EXISTING STAFF GAUGE
- C FORMER SOIL BORING
- LH-1/GMSB-7 EXISTING LEACHATE HEADWELL
- TP-14 FORMER TEST PIT
- T5-01 FORMER GEOPROBE BORING
- X RDB-01 PROPOSED GEOPROBE BORING

NOTES

1. BASE TOPOGRAPHY PROVIDED BY OAS, INC. OF SEYMOUR, INDIANA BASED ON AERIAL SURVEY DATED 3/30/2005. UPDATED TOPOGRAPHY FOR THE 12TH STREET LANDFILL WAS PROVIDED BY HOLLAND ENGINEERING, INC. SURVEY DATE: DECEMBER 6, 2007.
2. COORDINATES ARE MICHIGAN STATE PLANE-SOUTH ZONE. THE VERTICAL DATUM IS NGVD 29.
3. PROPERTY BOUNDARY BASED ON LEGAL DESCRIPTION PROVIDED BY U.S.EPA ON MARCH 30, 2004. COORDINATES FOR S 1/4 CORNER AND N 1/4 CORNER OF SECTION 24 AND NORTH BEARING USED TO PLOT PROPERTY LINE WERE PROVIDED BY HOLLAND ENGINEERING AND ARE BASED ON MICHIGAN STATE PLANE - SOUTH ZONE COORDINATES.
4. THE APPROXIMATE LIMITS OF VISIBLE RESIDUALS USING VISUAL CRITERIA WAS DERIVED FROM THE ALLIED PAPER, INC. /PORTAGE CREEK/KALAMAZOO RIVER SUPERFUND SITE REMEDIAL INVESTIGATION, TECHNICAL MEMORANDUM 8, (GERAGHTY AND MILLER, INC. MAY 31, 1994), AND REVISED BASED ON THE U.S. EPA'S 2003 PRECONSTRUCTION INVESTIGATION AND ON THE REMOVAL ACTIONS PERFORMED ALONG THE RIVERBANK IN 2007 AS PART OF THE EMERGENCY ACTION.

PROJECT: 12TH STREET LANDFILL
MULTI-AREA QAPP - REVISION 2
OTSEGO TOWNSHIP, MICHIGAN

SHEET TITLE: PROPOSED LANDFILL PREDESIGN INVESTIGATIONS

DRAWN BY: stormerl	SCALE: 1"=100'	PROJ. NO.	5117.02 \ Qapp
CHECKED BY: ECW		FILE NO.	PROPOSED LI.PLT
APPROVED BY: LEH	DATE PRINTED:	FIGURE	2-3
DATE: APRIL 2008			

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Reference Files
Ref. File 1 - J:\05117\02\Qapp\bmc120607.dgn(1) bmc
Ref. File 2 - J:\05117\02\Qapp\bmrmt.dgn (2) bmr
Ref. File 3 - J:\05117\02\Qapp\proposed.dgn (3) pro
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Section 3
Document Overview

This Multi-Area QAPP has been developed in accordance with the Uniform Federal Policy (UFP) QAPP guidance (U.S. EPA, 2005a), which is composed of a series of 37 worksheets, along with a series of attachments. The worksheets provide information on project management, project objectives, measurement and data acquisition, project assessment and oversight, and data review. The attachments include chain-of-custody records, laboratory certifications, laboratory quality assurance plans, and laboratory standard operating procedures (SOPs). In accordance with the U.S. EPA guidance, cross-referencing is utilized where applicable, in order to streamline the document. This includes providing references to planning documents that are companion documents to this Multi-Area QAPP.

Section 4
References

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QAPP Worksheet #1
Title and Approval Page

Site Name/Project Name:	Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Site Location:	Kalamazoo and Allegan Counties, including (but not limited to) 3 miles of Portage Creek and the Kalamazoo River from the City of Kalamazoo to Lake Michigan ▪ Operable Unit No. 4 - 12th Street Landfill ▪ Operable Unit No. 5 - Kalamazoo River ▪ Operable Unit No. 7 - Plainwell Mill
Document Title:	Multi-Area Quality Assurance Project Plan, Weyerhaeuser Company
Lead Organization:	U.S. EPA Region 5
Preparer's Name and Organizational Affiliation:	Kathryn Huibregtse, RMT, Inc.
Preparer's Contact Information:	150 North Patrick Blvd., Suite 180, Brookfield, WI 53045, 262.879.1212 kathy.huibregtse@rmtinc.com
Preparation Date:	Revision 00 - June 2007 Revision 01 - August 2007 Revision 02, Addendum 01 - September 2007 Revision 02, Addendum 02 - April 2008 Revision 02, Addendum 03 – September 2008
Investigative Organization's Project Coordinator For Emergency Response Activities in the Former Powerhouse Discharge Channel (Operable Unit No. 4):	Signature: _____ Jim Hutchens, RMT, Inc.
Investigative Organization's Project Coordinator For Emergency Response Activities for the 12th Street Landfill RD/RA (Operable Unit No. 4):	Signature: _____ Linda Hicken, RMT, Inc.
Investigative Organization's Project Coordinator For Investigative Activities at the Plainwell Mill (Operable Unit No. 7):	Signature: Jim Hutchens, RMT, Inc.
Investigative Organization's Project QA Manager:	Signature: _____ Kathryn Huibregtse, RMT, Inc.
Lead Organization's Program Manager for the 12th Street Landfill (Operable Unit No. 4):	Signature: _____ Michael Berkoff, U.S. EPA Region 5
Lead Organization's Program Manager for the Kalamazoo River (Operable Unit No. 5):	Signature: Jim Saric, U.S. EPA Region 5
Lead Organization's Program Manager for the Plainwell Mill (Operable Unit No. 7):	Signature: _____ Sam Chummar, U.S. EPA Region 5

Document Control Number: WEYCO-001

QAPP Worksheet #2
Identifying Information

Site Name/Project Name:	Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
Site Location:	Kalamazoo and Allegan Counties, including (but not limited to) 3 miles of Portage Creek and the Kalamazoo River from the city of Kalamazoo to Lake Michigan ▪ Operable Unit No. 4 - 12th Street Landfill ▪ Operable Unit No. 5 - Kalamazoo River ▪ Operable Unit No. 7 - Plainwell Mill
Site Number/Code:	MID006007306
Operable Unit:	Operable Units No. 4, 5, and 7
Contractor's Name:	NA
Contractor's Number:	NA
Contract Title:	NA
Work Assignment Number:	NA
Identify guidance used to prepare QAPP:	Uniform Federal Policy for Quality Assurance Project Plans, Manual VI (U.S. EPA, 2005a)
Identify regulatory program:	Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
Identify approval entity:	U.S. EPA Region 5
Indicate whether the QAPP is a generic or a project-specific QAPP:	This is a project-specific Multi-Area QAPP for emergency response activities at the 12th Street Landfill (Operable Unit No. 4), the Kalamazoo River banks (Operable Unit No. 5) adjacent to the Plainwell Mill, and for predesign investigations for the remedial design (RD) at the 12th Street Landfill (Operable Unit No. 4) and the investigation activities at the Plainwell Mill (Operable Unit No. 7). This QAPP will be updated as required for future work at the 12th Street Landfill Site and/or other sites.
List dates of scoping sessions that were held:	Emergency Activities for the 12th Street Landfill: May 8, 2007, and June 6, 2007, at U.S. EPA Region 5 offices in Chicago, Illinois Emergency Activities for the Plainwell Mill Banks: August 14, 2007, Submittal of conceptual design approach and follow-up discussions regarding U.S. EPA comments Predesign Investigations for the RD for the 12th Street Landfill: December 7, 2007, at U.S. EPA Region 5 offices in Chicago, Illinois, and on January 9, 2008, via conference call. Work Plan preparation and comment discussion for Phase 2 of Addendum No. 1 and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment: July 16, 2008, July 22, 2008 via conference call.

QAPP Worksheet #2 (continued)
Identifying Information

List dates and titles of QAPP documents written for previous site work, if applicable:	
Quality Assurance Project Plan, organization partners (stakeholders), and connection with lead organization:	***(Site-Wide QAPP)*** Blasland & Bouck Engineers, P.C. 1993. Quality Assurance Project Plan, June 1993. Prepared on behalf of the Kalamazoo River Study Group (KRSG). Approved by the U.S. EPA Region 5 and the Michigan Department of Natural Resources (MDNR). Other stakeholders included the Michigan Department of Environmental Quality (MDEQ), the United States Fish & Wildlife Service, and the National Oceanic & Atmospheric Administration.
Quality Assurance Project Plan, organization partners (stakeholders), and connection with lead organization: (continued):	***(Operable Unit No. 5)*** Arcadis BBL, 2007b. Draft Multi-Area Quality Assurance Project Plan, April 2007. Prepared on behalf of the Kalamazoo River Study Group, for the Time-Critical Removal Action (TCRA) and the Supplemental Remedial Investigation/Feasibility Study (SRI/FS). Approved by the U.S. EPA Region 5 on May 22, 2007.
	(Operable Unit No. 7) RMT, 2006. Draft Quality Assurance Project Plan for the Remedial Investigation/Feasibility Study – Plainwell Mill, September 2006. Prepared on behalf of Weyerhaeuser Company. Project stakeholders include the MDEQ and the City of Plainwell. The document is under review by the U.S. EPA, with response pending. This document is being replaced by this revision (Revision 02, Addendum 03 – September 2008) to the Multi-Area QAPP.
List data users:	Weyerhaeuser Company, U.S. EPA Region 5
Lead Organization's Program Manager:	**Operable Unit No. 4:** Michael Berkoff, U.S. EPA Region 5 Remedial Project Manager (RPM) **Operable Unit No. 5**: Jim Saric, U.S. EPA Region 5 RPM **Operable Unit No. 7:** Sam Chummar, U.S. EPA Region 5 RPM

Worksheet #2 (*continued*)
Identifying Information

REQUIRED QAPP ELEMENT(S) AND CORRESPONDING QAPP SECTION(S) (U.S. EPA, 2005a)	REQUIRED INFORMATION	CROSSWALK TO RELATED INFORMATION AND DOCUMENTS
Project Management and Objectives		
2.1 Title and Approval Page	▪ Title and Approval Page	Worksheet #1, Title and Approval Page
2.2 Document Format and Table of Contents	▪ Table of Contents ▪ QAPP Identifying Information	The Table of Contents is provided following the QAPP cover page. Worksheet #2, Identifying Information
2.2.1 Document Control Format		
2.2.2 Document Control Numbering System		
2.2.3 Table of Contents		
2.2.4 QAPP Identifying Information		
2.3 Distribution List and Project Personnel Sign-Off Sheet	▪ Distribution List ▪ Project Personnel Sign-Off Sheet	Worksheet #3, Distribution List; and Worksheets #4-1 and 4-2, Project Personnel Sign-Off Sheet
2.3.1 Distribution List		
2.3.2 Project Personnel Sign-Off Sheet		
2.4 Project Organization	▪ Project Organizational Chart ▪ Communication Pathways ▪ Personnel Responsibilities and Qualifications Table ▪ Special Personnel Training Requirements Table	Worksheets #5-1 through 5-3, Project Organization Charts; Worksheets #6-1 through 6-3, Communication Pathways; Worksheet #7, Personnel Responsibilities and Qualifications; and Worksheet #8, Special Personnel Training Requirements
2.4.1 Project Organization Chart		
2.4.2 Communication Pathways		
2.4.3 Personnel Responsibilities and Qualifications		
2.4.4 Special Training Requirements and Certification		
2.5 Project Planning/Problem Definition	▪ Project Planning Session ▪ Documentation (including Data Needs tables) ▪ Project Scoping Session Participants Sheet ▪ Problem Definition, Site History, and Background ▪ Site Maps (historical and current)	Worksheets #9-1 through 9-5, Project Scoping Session Participants Sheet; and Worksheets #10-1 through 10-4, Problem Definition-DQOs Site history and more detail concerning the project DQOs can be found in the companion documents to this Multi-Area QAPP. Site maps can be found on Figures 1-1, 1-2, and 2-1 through 2-3.
2.5.1 Project Planning (Scoping)		
2.5.2 Problem Definition, Site History, and Background		

Worksheet 2 (*continued*)
Identifying Information

REQUIRED QAPP ELEMENT(S) AND CORRESPONDING QAPP SECTION(S) (U.S. EPA, 2005a)	REQUIRED INFORMATION	CROSSWALK TO RELATED INFORMATION AND DOCUMENTS
Project Management and Objectives		
2.6 Project Quality Objectives and measurement Performance Criteria	▪ Site-Specific Project Quality Objectives (PQOs) ▪ Measurement Performance Criteria Table	Worksheets #11-1 through 11-4, Project Quality Objectives/Systematic Planning Process Statements; Worksheets #12-1 through 12-13, Measurement Performance Criteria
2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process		
2.6.2 Measurement performance Criteria		
2.7 Secondary Data Evaluation	▪ Sources of Secondary Data and Information ▪ Secondary Data Criteria and Limitations Table	Worksheets #13-1 and 13-2, Secondary Data Criteria and Limitations
2.8 Project Overview and Schedule	▪ Summary of Project Tasks ▪ Reference Limits and Evaluation Table ▪ Project Schedule/Timeline Table	Worksheet #14, Summary of Project Tasks; Worksheets #15-1 through 15-4, Reference Limits and Evaluation; and Worksheets #16-1 through 16-4, Project Schedules/Timelines
2.8.1 Project Overview		
2.8.2 Project Schedule		

Worksheet 2 (*continued*)
Identifying Information

REQUIRED QAPP ELEMENT(S) AND CORRESPONDING QAPP SECTION(S) (U.S. EPA, 2005a)	REQUIRED INFORMATION	CROSSWALK TO RELATED INFORMATION AND DOCUMENTS
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 Container Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	▪ 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	Worksheet #17-1 through 17-3, Sampling Design and Rationale; Worksheets #18-1 through 18-3, Sampling Locations and Methods/SOP Requirements; Worksheet #19, Analytical SOP Requirements (sample containers, preservation, and holding times); Worksheet #20, Field Quality Control Sample Summary Worksheet #21, Project Sampling SOP Reference; and Worksheet #22, Field Equipment Calibration, Maintenance, Testing, and Inspection The laboratory SOPs can be found in Attachment 1-5. More details concerning the sampling design and rationale and the field sampling procedures can be found in the companion documents to this Multi-Area QAPP.
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	▪ Analytical SOPs ▪ Analytical SOP References Table ▪ Analytical Instrument Calibration Table ▪ Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Worksheet #23, Analytical SOP References; Worksheet #24, Analytical Instrument Calibration; and Worksheet #25, Analytical Instrument and Equipment Maintenance, Testing, and Inspection The laboratory SOPs can be found in Attachment 1-5.

Worksheet 2 (*continued*)
Identifying Information

REQUIRED QAPP ELEMENT(S) AND CORRESPONDING QAPP SECTION(S) (U.S. EPA, 2005a)	REQUIRED INFORMATION	CROSSWALK TO RELATED INFORMATION AND DOCUMENTS
Measurement/Data Acquisition (*continued*)		
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures	▪ Sample Collection Documentation Handling, Tracking, and Custody SOPs ▪ Sample Container Identification ▪ Sample Handling Flow Diagram ▪ Example Chain-of-Custody Record and Seal	Worksheet #27, Sample Custody Requirements More details concerning the field sampling procedures can be found in the companion documents to this Multi-Area QAPP. Example COC forms can be found in Attachment 1-3.
3.3.1 Sample Collection Documentation		
3.3.2 Sample Handling and Tracking System		
3.3.3 Sample Custody		
3.4 Quality Control Samples	▪ QC Samples Table ▪ Screening/Confirmatory Analysis Decision Tree	Worksheets #28-1 through 28-15, Present QC sample information for project analysis
3.4.1 Sampling Quality Control Samples		
3.4.2 Analytical Quality Control Samples		
3.5 Data Management Tasks	▪ Project Documents and Records Table ▪ Analytical Services Table ▪ Data Management SOPs	Worksheet #29, Project Documents and Records, and Worksheet #30, Analytical Services
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		
4.1 Assessments and Response Actions	▪ Assessments and Response Actions ▪ Planned Project Assessments Table ▪ Audit Checklists ▪ Assessment Findings and Corrective Action Responses Table	Worksheets #31-1 through 31-3, Planned Project Assessments, and Worksheets #32-1 and 32-2, Assessment Findings and Corrective Action Responses The laboratory Quality Assurance Manual and Laboratory Policies and Guidelines documents can be found in Attachments 1-4 and 1-6, respectively.
4.1.1 Planned Assessments		
4.1.2 Assessment Findings and Corrective Action Responses		
4.2 QA Management Reports	▪ QA Management Reports Table	Worksheet #33, QA Management Reports
4.3 Final Project Report		

Worksheet 2 (*continued*)
Identifying Information

REQUIRED QAPP ELEMENT(S) AND CORRESPONDING QAPP SECTION(S) (U.S. EPA, 2005a)	REQUIRED INFORMATION	CROSSWALK TO RELATED INFORMATION AND DOCUMENTS
Data Review		
5.1 Overview		
5.2 Data Review Steps	▪ Verification (Step I) Process Table ▪ Validation (Steps IIa and IIb) Process Table ▪ Validation (Steps IIa and IIb) Summary Table ▪ Usability Assessment	Worksheet #34, Verification (Step 1) Process; Worksheet #35, Validation (Step I) Process; Worksheet #36, Validation (Steps IIa and IIb) Summary; and Worksheet #37, Usability Assessment.
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		
5.3 Streamlining Data Review	▪ None	NA
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		

QAPP Worksheet #3
Distribution List

QAPP RECIPIENTS	TITLE	ORGANIZATION	TELEPHONE NUMBER	E-MAIL ADDRESS	DOCUMENT CONTROL NUMBER
Michael Berkoff (2 copies)	Remedial Project Manager	U.S. EPA, Region 5	312.353.8983	berkoff.michael@epa.gov	WEYCO-001
Sam Chummar (2 copies)	Remedial Project Manager	U.S. EPA, Region 5	312.886.1434	Chummar.Sam@epamail.epa.gov	WEYCO-001
Jim Saric	Remedial Project Manager	U.S. EPA, Region 5	312. 886.0992	Saric.james@epa.gov	WEYCO-001
Jennifer Hale	Environmental Manager	Weyerhaeuser Company	253.924.3746	jennifer.hale@weyerhaeuser.com	WEYCO-001
Martin Lebo	Senior Aquatic Scientist	Weyerhaeuser Company	252.633.7511	martin.lebo@weyerhaeuser.com	WEYCO-001
Jim Hutchens	Senior Project Manager	RMT	262.879.1212	james.hutchens@rmtinc.com	WEYCO-001
Linda Hicken	Senior Project Manager	RMT	608.662.5307	linda.hicken@rmtinc.com	WEYCO-001
Kathy Huibregtse	Principal-In-Charge	RMT	262.879.1212	kathy.huibregtse@rmtinc.com	WEYCO-001
Jennifer Overvoorde	Field Sampling Coordinator and Health and Safety Officer	RMT	616.975.5415	jennifer.overvoorde@rmtinc.com	WEYCO-001
Jeff Macri	Construction Manager	RMT	317.490.2865 (mobile)	jeff.macri@rmtinc.com	WEYCO-001
Mike Amstadt	Senior Project Engineer	RMT	608.662.5271	mike.amstadt@rmtinc.com	WEYCO-001

QAPP Worksheet #3 (continued)
Distribution List

QAPP RECIPIENTS	TITLE	ORGANIZATION	TELEPHONE NUMBER	E-MAIL ADDRESS	DOCUMENT CONTROL NUMBER
Tom Stolzenburg	Data QA Manager	RMT	608.662.5287	tom.stolzenburg@rmtinc.com	WEYCO-001
Cheryl Shaw	Database Manager	RMT	608.662.5117	cheryl.shaw@rmtinc.com	WEYCO-001
Dennis Catalano	Laboratory QA/QC Manager	WATS	253.924.6242	dennis.catalano@weyerhaeuser.com	WEYCO-001

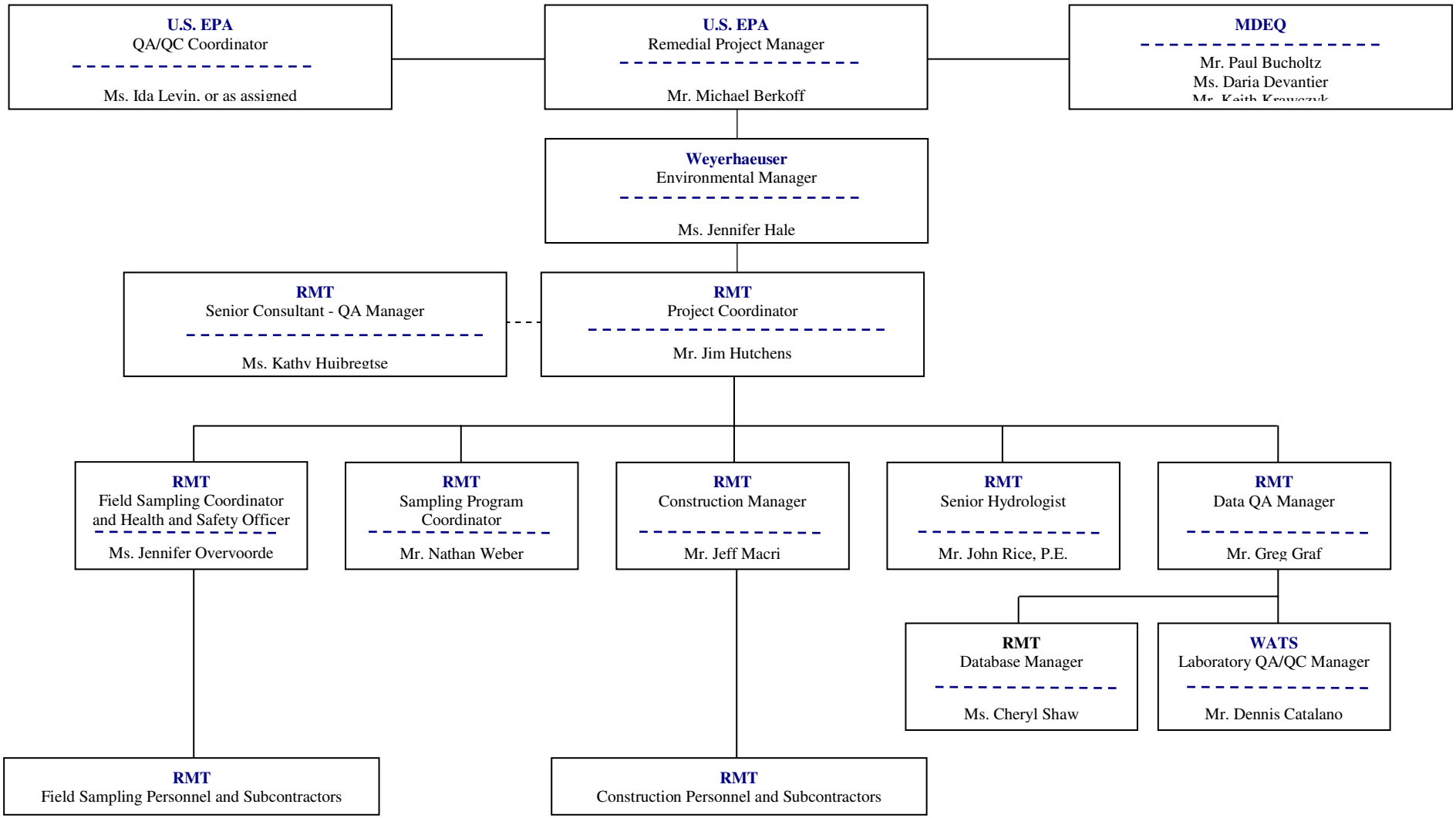
QAPP Worksheet #4-1
Project Personnel Sign-Off Sheet (RMT)

PROJECT PERSONNEL	TITLE	TELEPHONE NUMBER	SIGNATURE	DATE QAPP READ
Jim Hutchens	Senior Project Manager	262.879.1212		
Linda Hicken	Senior Project Manager	608.662.5307		
Kathy Huibregtse	Principal-In-Charge	262.879.1212		
Jennifer Overvoorde	Field Sampling Coordinator and Health and Safety Officer	616.975.5415		
Jeff Macri	Construction Manager	317.490.2865		
Mike Amstadt	Senior Project Engineer	608.662.5271		
Tom Stolzenburg	Data QA Manager	608.662.5287		
Cheryl Shaw	Database Manager	608.662.5117		

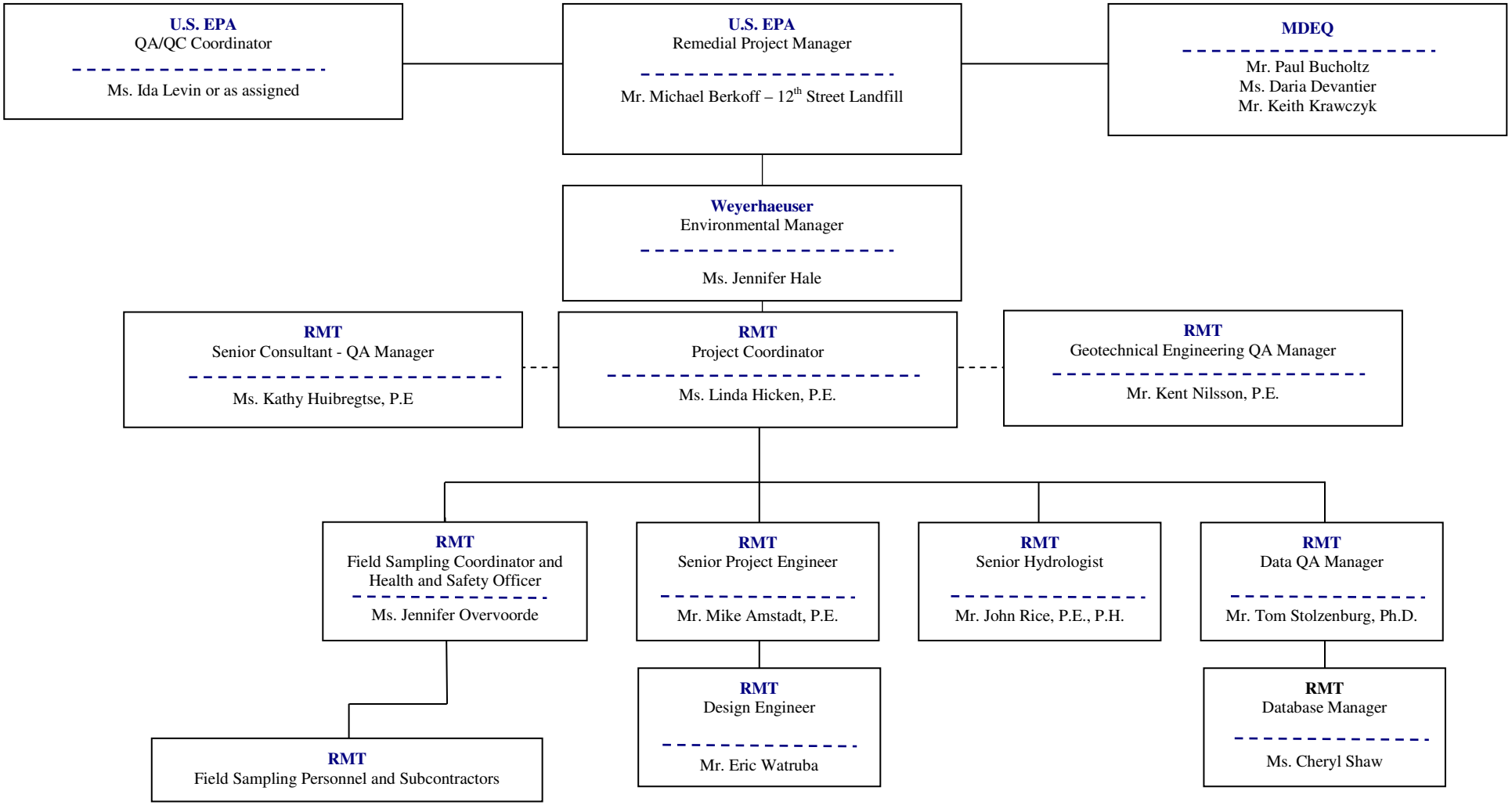
QAPP Worksheet #4-2
Project Personnel Sign-Off Sheet (WATS)

PROJECT PERSONNEL	TITLE	TELEPHONE NUMBER	SIGNATURE	DATE QAPP READ
Dennis Catalano	Laboratory QA/QC Manager	253.924.6242		

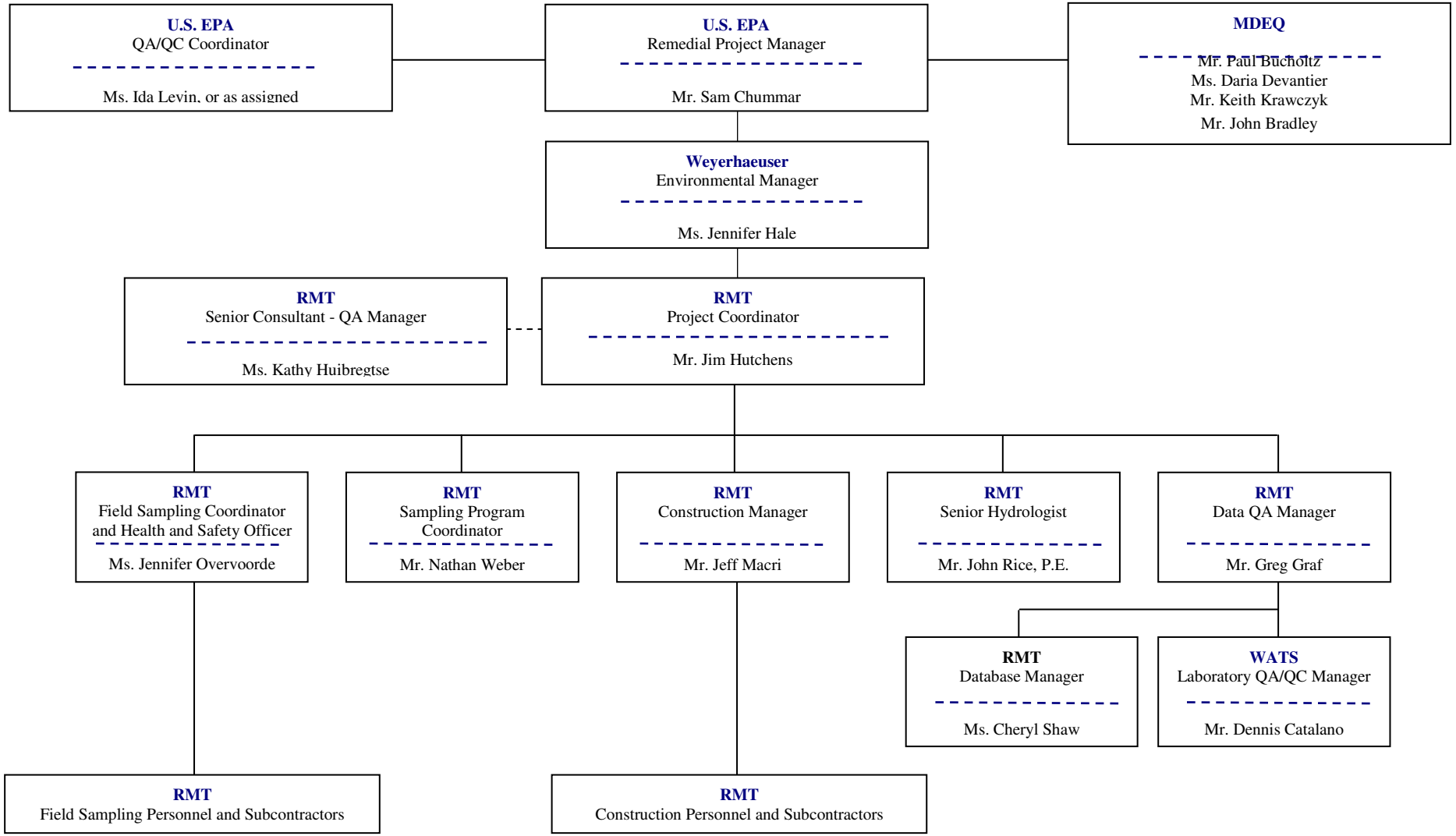
QAPP Worksheet #5-1
Project Organizational Chart
(Emergency Response Activities for the 12th Street Landfill and the Plainwell Mill Banks)



QAPP Worksheet #5-2
Project Organizational Chart
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)



QAPP Worksheet #5-3
Project Organizational Chart
(Phase 2 of Addendum No. 1 [Test Pits] and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)



QAPP Worksheet #6-1
Communication Pathways
(Emergency Response Activities For the 12th Street Landfill and the Plainwell Mill Banks)

COMMUNICATION DRIVERS	RESPONSIBLE ENTITY	NAME	TELEPHONE NUMBER	PROCEDURE (timing, pathways, etc.)
Emergency Response Activities in the Former Powerhouse Discharge Channel (Operable Unit No. 4)				
Serve as Point-of-Contact with U.S. EPA Remedial Project Manager (RPM)	Environmental Manager	Jennifer Hale, Weyerhaeuser Company	253.924.3746	Jennifer Hale will provide project submittals to Michael Berkoff, U.S. EPA, in accordance with the schedule provided on Worksheet No. 16 of this QAPP.
Manage all project phases	Project Coordinator	Jim Hutchens, RMT	262.879.1212	Jim Hutchens will be the RMT liaison to the U.S. EPA for emergency response activities in the former powerhouse discharge channel at the 12th Street Landfill site.
Coordinate field program	Field Sampling Coordinator and Health and Safety Officer	Jennifer Overvoorde, RMT	616.975.5415	Jennifer Overvoorde will provide Jim Hutchens with an update of field sampling activities, including related questions or problems, by telephone or e-mail at the end of each business day. Jennifer Overvoorde will also report any QA/QC problems related to field measurements to the Data QA Manager (Greg Graf) by telephone or e-mail by the end of the same business day.
Provide daily progress reports – field sampling	Field Sampling Coordinator and Health and Safety Officer	Jennifer Overvoorde, RMT	616.975.5415	Jennifer Overvoorde will provide Jim Hutchens with written daily progress reports, including field records, sampling logs, Chain-of-Custody Records, and any other pertinent information by e-mail or fax within 2 business days.
Notify Jim Hutchens and the Data QA Manager of any QAPP changes in the field	Field Sampling Coordinator and Health and Safety Officer	Jennifer Overvoorde, RMT	616.975.5415	Jennifer Overvoorde will notify Jim Hutchens and the Data QA Manager (Greg Graf) on the same business day of any changes to the QAPP made in the field, including the reasons, by telephone or e-mail.

QAPP Worksheet #6-1 (*continued*)
Communication Pathways
(Emergency Response Activities for the 12th Street Landfill and the Plainwell Mill Banks)

COMMUNICATION DRIVERS	RESPONSIBLE ENTITY	NAME	TELEPHONE NUMBER	PROCEDURE (timing, pathways, etc.)
Coordinate construction activities	Construction Manager	Jeff Macri, RMT	317.490.2865	Jeff Macri will provide Jim Hutchens with an update of construction activities, including related questions or problems, by telephone or e-mail at the end of each business day.
Daily progress reports – construction activities	Construction Manager	Jeff Macri, RMT	317.490.2865	Jeff Macri will provide Jim Hutchens with written daily progress reports, including field records and any other pertinent information, by e-mail or fax within 2 business days.
Reporting lab data quality issues	Laboratory QA/QC Manager	Dennis Catalano, WATS	253.924.6242	Dennis Catalano will notify the Data QA Manager (Greg Graf) with any QA/QC issues within 2 business days.
Field and analytical corrective actions	Data QA Manager	Greg Graf, RMT	608.662.5306	Greg Graf will determine the need for corrective action for field and analytical issues, in conjunction with the Project Coordinator, the Field Sampling Coordinator, or the Laboratory QA/QC Manager, as appropriate.
Release of final analytical data	Data QA Manager	Greg Graf, RMT	608.662.5306	No final analytical data can be released until validation is complete and Greg Graf has approved the release.
QAPP amendments	Data QA Manager	Greg Graf, RMT	608.662.5306	Any major changes to the QAPP must be approved by Greg Graf, the Project Coordinator, and the U.S. EPA before the changes can be implemented.

QAPP Worksheet #6-2
Communication Pathways
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)

COMMUNICATION DRIVERS	RESPONSIBLE ENTITY	NAME	TELEPHONE NUMBER	PROCEDURE (timing, pathways, etc.)
Serve as Point-of-Contact with U.S. EPA Remedial Project Manager (RPM)	Environmental Manager	Jennifer Hale, Weyerhaeuser Company	253.924.3746	Jennifer Hale will provide project submittals to the U.S. EPA RPM associated with the applicable OU, in accordance with the schedule provided on Worksheet No. 16-2 of this QAPP.
Manage all project phases	Project Coordinator	Linda Hicken, RMT	608.662.5307	Linda Hicken will be the RMT liaison to the U.S. EPA for the remedial design for the 12th Street Landfill site.
Coordinate field program	Field Sampling Coordinator and Health and Safety Officer	Jennifer Overvoorde, RMT	616.975.5415	Jennifer Overvoorde will provide Linda Hicken with an update of field sampling activities, including related questions or problems, by telephone or e-mail at the end of each business day. Jennifer Overvoorde will also report any QA/QC problems related to field measurements to the Data QA Manager (Tom Stolzenburg) by telephone or e-mail by the end of the same business day.
Provide daily progress reports – field sampling	Field Sampling Coordinator and Health and Safety Officer	Jennifer Overvoorde, RMT	616.975.5415	Jennifer Overvoorde will provide Linda Hicken with written daily progress reports, including field records and any other pertinent information by e-mail or fax within 2 business days.
Notify Linda Hicken and the Data QA Manager of any QAPP changes in the field	Field Sampling Coordinator and Health and Safety Officer	Jennifer Overvoorde, RMT	616.975.5415	Jennifer Overvoorde will notify Linda Hicken and the Data QA Manager (Tom Stolzenburg) on the same business day of any changes to the QAPP made in the field, including the reasons, by telephone or e-mail.

QAPP Worksheet #6-2 (*continued*)
Communication Pathways
Predesign Investigations for the Remedial Design for the 12th Street Landfill

COMMUNICATION DRIVERS	RESPONSIBLE ENTITY	NAME	TELEPHONE NUMBER	PROCEDURE (timing, pathways, etc.)
Field corrective actions	Senior Project Engineer	Mike Amstadt, RMT	608.662.5271	Mike Amstadt will determine the need for corrective action for field issues, in conjunction with the Project Coordinator, or the Field Sampling Coordinator, as appropriate.
QAPP amendments	Data QA Manager	Tom Stolzenburg, RMT	608.662.5306	Any major changes to the QAPP must be approved by Tom Stolzenburg, the Project Coordinator, and the U.S. EPA before the changes can be implemented.

QAPP Worksheet #6-3
Communication Pathways
(Phase 2 of Addendum No. 1 [Test Pits] and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)

COMMUNICATION DRIVERS	RESPONSIBLE ENTITY	NAME	TELEPHONE NUMBER	PROCEDURE (timing, pathways, etc.)
Emergency Response Activities in the Former Powerhouse Discharge Channel (Operable Unit No. 4)				
Serve as Point-of-Contact with U.S. EPA Remedial Project Manager (RPM)	Environmental Manager	Jennifer Hale, Weyerhaeuser Company	253.924.3746	Jennifer Hale will provide project submittals to Sam Chummar, U.S. EPA, in accordance with the schedule provided on Worksheet No. 16 of this QAPP.
Manage all project phases	Project Coordinator	Jim Hutchens, RMT	262.879.1212	Jim Hutchens will be the RMT liaison to the U.S. EPA for emergency response activities at the Plainwell Mill property.
Coordinate field program	Field Sampling Coordinator and Health and Safety Officer	Jennifer Overvoorde, RMT	616.975.5415	Jennifer Overvoorde will provide Jim Hutchens with an update of field sampling activities, including related questions or problems, by telephone or e-mail at the end of each business day. Jennifer Overvoorde will also report any QA/QC problems related to field measurements to the Data QA Manager (Greg Graf) by telephone or e-mail by the end of the same business day.
Provide daily progress reports – field sampling	Field Sampling Coordinator and Health and Safety Officer	Jennifer Overvoorde, RMT	616.975.5415	Jennifer Overvoorde will provide Jim Hutchens with written daily progress reports, including field records, sampling logs, Chain-of-Custody Records, and any other pertinent information by e-mail or fax within 2 business days.
Notify Jim Hutchens and the Data QA Manager of any QAPP changes in the field	Field Sampling Coordinator and Health and Safety Officer	Jennifer Overvoorde, RMT	616.975.5415	Jennifer Overvoorde will notify Jim Hutchens and the Data QA Manager (Greg Graf) on the same business day of any changes to the QAPP made in the field, including the reasons, by telephone or e-mail.

QAPP Worksheet #6-3 (*continued*)
Communication Pathways
(Phase 2 of Addendum No. 1 [Test Pits] and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)

COMMUNICATION DRIVERS	RESPONSIBLE ENTITY	NAME	TELEPHONE NUMBER	PROCEDURE (timing, pathways, etc.)
Coordinate construction activities	Construction Manager	Jeff Macri, RMT	317.490.2865	Jeff Macri will provide Jim Hutchens with an update of construction activities, including related questions or problems, by telephone or e-mail at the end of each business day.
Daily progress reports – construction activities	Construction Manager	Jeff Macri, RMT	317.490.2865	Jeff Macri will provide Jim Hutchens with written daily progress reports, including field records and any other pertinent information, by e-mail or fax within 2 business days.
Reporting lab data quality issues	Laboratory QA/QC Manager	Dennis Catalano, WATS	253.924.6242	Dennis Catalano will notify the Data QA Manager (Greg Graf) with any QA/QC issues within 2 business days.
Field and analytical corrective actions	Data QA Manager	Greg Graf, RMT	608.662.5306	Greg Graf will determine the need for corrective action for field and analytical issues, in conjunction with the Project Coordinator, the Field Sampling Coordinator, or the Laboratory QA/QC Manager, as appropriate.
Release of final analytical data	Data QA Manager	Greg Graf, RMT	608.662.5306	No final analytical data can be released until validation is complete and Greg Graf has approved the release.
QAPP amendments	Data QA Manager	Greg Graf, RMT	608.662.5306	Any major changes to the QAPP must be approved by Greg Graf, the Project Coordinator, and the U.S. EPA before the changes can be implemented.

QAPP Worksheet #7
Personnel Responsibilities and Qualifications

NAME	PROJECT ROLE	ORGANIZATIONAL AFFILIATION	EDUCATION AND EXPERIENCE QUALIFICATIONS
Michael Berkoff Sam Chummar	Remedial Project Manager - 12th Street Landfill Remedial Project Manager - Plainwell Mill	U.S. EPA, Region 5	Designated as the U.S. EPA's Project Coordinators
Jennifer Hale	Environmental Manager	Weyerhaeuser	B.S. in ecology and evolutionary biology, with a combined minor in chemistry, physics and mathematics, University of Arizona, 1998 Over 5 years of experience
Kathryn Huibregtse, P.E.	Senior Consultant - QA/QC Manager	RMT	B.S. chemical engineering, University of Wisconsin (UW)-Madison, 1974 Over 28 years of experience
Jim Hutchens, P.E.	Project Coordinator	RMT	B.S. mining engineering, University of Wisconsin (UW)-Platteville, 1981 Over 20 years of experience
Linda Hicken, P.E.	Project Coordinator	RMT	MBA, University of Southern California, 1983 B.S. chemical engineering, University of Michigan-Ann Arbor, 1980 28 years of experience
Nathan Weber	Sampling Program Coordinator	RMT	B.S. chemical engineering, University of Wisconsin (UW)-Madison, 2003 Over 5 years of experience
Eric Watruba	Sampling Program Coordinator/Design Engineer	RMT	B.S. geological engineering, University of Wisconsin (UW)-Madison, 2004 B.S. geology and geophysics, University of Wisconsin(UW)-Madison, 2004 3 years of experience
Jeff Macri	Construction Manager	RMT	MBA, University of Indianapolis, 1996 B.S. chemistry, Butler University, 1985 20 years of experience
John Rice, P.E., P.H.	Senior Hydrologist	RMT	M.S. civil environmental engineering, University of Wisconsin (UW)-Madison, 1984 B.S civil environmental engineering, University of Wisconsin (UW)-Madison, 1982 Over 23 years of experience
Mike Amstadt, P.E.	Senior Project Engineer	RMT	B.S. civil and environmental engineering, University of Wisconsin (UW)-Madison, 1990 17 years of experience
Tom Stolzenburg, Ph.D.	Data QA Manager	RMT	Ph.D., civil and environmental engineering, water chemistry program, University of Wisconsin - Madison, 1979, B.S., resource management and biology, UW-Stevens Point, 1974. Over 28 years of experience.

QAPP Worksheet #7 (continued)
Personnel Responsibilities and Qualifications

NAME	PROJECT ROLE	ORGANIZATIONAL AFFILIATION	EDUCATION AND EXPERIENCE QUALIFICATIONS
Kent Nilsson, P.E.	Geotechnical Engineering QA Manager	RMT	B.S. civil engineering, Brigham Young University, 1983 M.E.M. civil engineering, Brigham Young University, 1984 23 years experience
Jennifer Overvoorde	Field Coordinator and Health and Safety Officer	RMT	B.A. geology, Calvin College, 1998 Over 10 years of experience
Cheryl Shaw	Database Manager	RMT	Chemistry course work, University of Wisconsin (UW)-Madison 8 years of experience
Dennis Catalano	Laboratory QA/QC Manager	WATS	B.S. biology with chemistry minor, University of Dayton, 1977 30 years of experience

QAPP Worksheet #7 (*continued*)
Personnel Responsibilities and Qualifications

U. S. EPA

Project Coordinator

- Serve as the primary point-of-contact for the U.S. EPA.
- Review and approve project submittals.
- Coordinate with the Michigan DEQ and other regulatory agencies as needed.
- Monitor project activities.

QA/QC Coordinator

- Review and approve the QAPP.
- Review and approve relevant portions of submitted documents to ensure compliance with the QAPP.
- Coordinate audits, as necessary.

WEYERHAEUSER COMPANY

Environmental Manager

- Serve as the primary point-of-contact between Weyerhaeuser Company and the U.S. EPA.
- Coordinate communications between Weyerhaeuser, RMT**,** the U.S. EPA, and other parties.
- Review draft project deliverables prior to their submittal to the U.S. EPA.

RMT

Project Coordinator

- Serve as the primary point-of-contact for RMT.
- Communicate with Weyerhaeuser Company, the U.S. EPA, and other project stakeholders.
- Establish and communicate project milestones and schedules.
- Coordinate and review RMT work products.
- Direct RMT team members.

QAPP Worksheet #7 (*continued*)
Personnel Responsibilities and Qualifications

Senior Consultant – QA Manager

- Provide overall quality assurance, including a review of RMT's performance on the project.
- Review project progress and RMT work products for compliance with project objectives and requirements.
- Provide input regarding Superfund procedures and compliance with the National Contingency Plan (NCP).

Field Sampling Coordinator and Health and Safety Officer

- Coordinate environmental staff who will be measuring and recording field parameters, collecting samples, and documenting construction activities, to ensure compliance with this QAPP.
- Order sample bottles from the laboratory.
- Prepare reports pertaining to relevant field activities.
- Maintain field and laboratory files, including project documentation, sample logs, and calculations; and provide complete copies to the RMT Project Coordinator.
- Provide on-site Health and Safety Orientation to all RMT staff and subcontractors, and monitor site activities for compliance with the project Health and Safety Plan.
- Conduct field audits, as necessary.

Sampling Program Coordinator/Design Engineer

- Review existing data, project objectives, and regulatory guidance to assist in the development of project sampling protocols.
- Review and summarize the results of data collected during project implementation, and make preliminary recommendations to the RMT Project Coordinator regarding the results (*e.g.*, whether additional data collection or field modifications appear to be necessary).

Construction Manager

- Coordinate and direct subcontractors and RMT personnel who are performing construction work (*e.g.*, site preparation, excavation).
- Assist Field Sampling Coordinator with Health and Safety Orientations for construction-related activities.
- Maintain field records related to construction activities, and provide complete copies to the RMT Project Coordinator.

Senior Hydrologist

- Provide technical input during the planning, design, and implementation stages of the project.
- Review the technical aspects of RMT's submittals.
- Serve as the professional engineer of record for work related to hydrology.

QAPP Worksheet #7 (*continued*)
Personnel Responsibilities and Qualifications

Senior Project Engineer

- Provide technical input during the planning, design, and implementation stages of the project.
- Review the technical aspects of RMT's submittals.
- Serve as the professional engineer of record for work related to landfill design.

Geotechnical Engineering QA Manager

- Provide technical input during the planning, design, and implementation stages of the project.
- Review the technical aspects of RMT's submittals.

Data QA Manager

- Serve as the primary point-of-contact with the laboratory.
- Coordinate field QA/QC procedures with the Field Coordinator (*e.g.*, calibrations for field analytical measurements, and field audits, as necessary), and review pertinent field records for compliance with this Quality Assurance Project Plan (QAPP).
- Review laboratory QA/QC procedures and documentation, as provided in data deliverables.
- Review data for compliance with the Data Quality Objectives (DQOs) for the project.
- Oversee data validation activities and the preparation of QA/QC reports by the 3rd party data validator.

Database Manager

- Establish and maintain a project database.
- Import electronic data deliverables (EDDs) provided by the laboratory into the project database.
- Perform a QA/QC check of imported data versus the hard copy data.
- Transcribe field and non-EDD laboratory data into the project database, as necessary.
- Tabulate data for end users.

ANALYTICAL LABORATORY

Laboratory QA/QC Manager

- Serve as the primary point-of-contact for the laboratory.
- Oversee preparation of analytical reports.
- Verify that the laboratory meets the QA/QC standards specified in this QAPP.
- Supervise in-house chain-of-custody.
- Perform laboratory audits, in accordance with this QAPP.

QAPP Worksheet #8
Special Personnel Training Requirements

PROJECT FUNCTION	SPECIALIZED TRAINING - TITLE OR DESCRIPTION OF COURSE	TRAINING PROVIDER	TRAINING DATE	PERSONNEL/ GROUPS RECEIVING TRAINING	PERSONNEL TITLES/ ORGANIZATIONAL AFFILIATION	LOCATION OF TRAINING RECORDS/ CERTIFICATES
Field activities	40-hour HAZWOPER and 8-hour HAZWOPER refresher	Certified training professionals	8-hour refresher within previous year	Field personnel	RMT personnel	RMT project offices
Confined Space Entry	Confined space training and testing, if applicable	Certified training professionals	4-hour training	Field personnel	RMT personnel	RMT project offices
Analytical chemistry	No state laboratory certification program in Michigan(1)	N/A	N/A	N/A	Weyerhaeuser Analysis and Testing Services (WATS) Weyerhaeuser Company Mail Stop: WTC2F25 32901 Weyerhaeuser Way Federal Way, WA 98001 Dennis Catalano 253.924.6242	N/A

Footnote:

(1) WATS is certified by the State of Washington.

QAPP Worksheet #9-1
Project Scoping Session Participants Sheet (May 8, 2007)

Project Name: Emergency Response Activities for Operable Unit No. 4

Projected Date(s) of Sampling: 2007 **Site Name:** Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

RMT Project Managers: Jim Hutchens (former powerhouse discharge channel) and Linda Hicken (12th Street Landfill) **Location of Scoping Session:** U.S. EPA Region 5, Chicago, Illinois

Date of Session: May 8, 2007

Scoping Session Purpose: To discuss the administrative mechanism for implementing actions at the 12th Street Landfill O.U. to accommodate the KRSG's Time Critical Removal Action, and to discuss the objectives and scope of a geotechnical investigation necessary to design a stable sideslope of the landfill along the river to accommodate the TCRA.

NAME	TITLE	AFFILIATION	TELEPHONE NUMBER	E-MAIL ADDRESS	PROJECT ROLE
Eileen Furey	Associate Regional Counsel	U.S. EPA, Region 5	312.886.7950	furey.eileen@epa.gov	Counsel for U.S. EPA
Tim Prendiville	Remedial Project Manager	U.S. EPA, Region 5	312.886.5122	prendiville.timothy@epa.gov	(Outgoing) Project Coordinator for 12th Street Landfill
Michael Berkoff	Remedial Project Manager	U.S. EPA, Region 5	312.353.8983	berkoff.michael@epa.gov	(Incoming) Project Coordinator for 12th Street Landfill
Ida Levin	Superfund Division Quality Assurance - Team Leader	U.S. EPA, Region 5	312.886.6254	levin.ida@epa.gov	U.S. EPA QA/QC Coordinator
Sam Borries	On-Scene Coordinator	U.S. EPA, Region 5	312.353.8360	borries.samuel@epa.gov	On-Scene Coordinator for the TCRA
Daria Devantier (by telephone)	Superfund Section - Specialized Sampling Unit - Team Leader	MDEQ	517.373.8436	devantid@michigan.gov	MDEQ Regulatory Support
Paul Bucholtz (by telephone)	Superfund Section - Specialized Sampling Unit - Project Manager	MDEQ	517.373.8174	bucholtp@michigan.gov	MDEQ Project Manager
Keith Krawczyk (by telephone)	Superfund Section - Specialized Sampling Unit - Project Manager	MDEQ	517.335.1104	krawczyk@michigan.gov	MDEQ Regulatory Support

QAPP Worksheet #9-1 (*continued*)
Project Scoping Session Participants Sheet (May 8, 2007)

NAME	TITLE	AFFILIATION	TELEPHONE NUMBER	E-MAIL ADDRESS	PROJECT ROLE
Jennifer Hale	Environmental Manager	Weyerhaeuser Company	253.924.3746	jennifer.hale@weyerhaeuser.com	Project Coordinator for Weyerhaeuser
Martin Lebo, Ph.D (by telephone)	Senior Aquatic Scientist	Weyerhaeuser Company	252.633.7511	martin.lebo@weyerhaeuser.com	Weyerhaeuser technical review
Joe Jackowski (by telephone)	Senior Legal Counsel	Weyerhaeuser Company	253.924.3461	joe.jackowski@weyerhaeuser.com	Counsel for Weyerhaeuser
Mark Schneider (by telephone)	Attorney at Law	Perkins Coie	206.359.8627	mwschneider@perkinscoie.com	Outside Counsel for Weyerhaeuser
Jeff Keiser	Project Manager	CH2M Hill	414.847.0382	jeff.keiser@ch2m.com	Oversight Contractor to the U.S. EPA
Linda Hicken	Senior Project Manager	RMT, Inc.	608.662.5307	linda.hicken@rmtinc.com	RMT Project Manager for 12th Street Landfill RD/RA
Jim Hutchens	Senior Project Manager	RMT, Inc.	262.879.1212	james.hutchens@rmtinc.com	RMT Project Manager for Emergency Actions in the former powerhouse discharge channel
Kathy Huibregtse	Principal-In-Charge	RMT, Inc.	262.879.1212	kathy.huibregtse@rmtinc.com	Senior Consultant - QA Manager

QAPP Worksheet #9-1 (*continued*)
Project Scoping Session Participants Sheet (May 8, 2007)

Comments/Decisions:

- Michael Berkoff is the new U.S. EPA Remedial Project Manager for all the landfill operable units on the Kalamazoo River. Weyerhaeuser will communicate directly with him going forward.
- The U.S. EPA decided that the removal of sediment in the former powerhouse discharge channel and work on the 12th Street Landfill necessary to accommodate the Time Critical Removal Action (TCRA) by the Kalamazoo River Study Group (KRSG) should proceed as an emergency response action, as defined under Section XVII, paragraph 67 of Weyerhaeuser's Consent Decree with the U.S. EPA.
- The Statement of Work for the Remedial Design and Remedial Action for the 12th Street Landfill should ultimately be modified to reflect the change in the order of implementing the required tasks.
- Weyerhaeuser can either prepare its own QAPP for the emergency response activities, or have Arcadis BBL (on behalf of the KRSG) collect samples under the KRSG's QAPP, once it is approved by the U.S. EPA.
- Work schedules need to be coordinated with the KRSG. The KRSG wants to remove the coffer dam this fall to avoid ice damage and to possibly proceed with Phase 2.

Action Items:

- Weyerhaeuser is to submit a document describing the Data Quality Objectives (DQOs) and the scope of a geotechnical investigation of the reported berm along the riverfront of the landfill. Weyerhaeuser is to also submit a Health and Safety Plan for the geotechnical investigation.
- Weyerhaeuser is to review the U.S. EPA's new format for QAPPs and the KRSG's QAPP for the TCRA, and decide on which format to use for the QAPP for the emergency response activities.

Consensus Decisions:

- Weyerhaeuser can submit a QAPP for the emergency response activities in either the old or the new format.

QAPP Worksheet #9-2
Project Scoping Session Participants Sheet (June 6, 2007)

Project Name: Emergency Response Activities for the 12th Street Landfill (Operable Unit No. 4)

Projected Date(s) of Sampling: 2007 **Site Name:** Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

Project Manager: Jim Hutchens, RMT **Location of Scoping Session:** U.S. EPA Region 5 offices, Chicago, Illinois

Date of Session: June 6, 2007

Scoping Session Purpose: Update meeting on former powerhouse discharge channel emergency actions. Specific purpose: to present an overview of the emergency response actions being designed; to review initial results from the geotechnical evaluation; to discuss bathymetric and visual sediment assessment data needs; to refine communication procedures, and to update the construction schedule. New team member from the U.S. EPA was introduced.

NAME	TITLE	AFFILIATION	TELEPHONE NUMBER	E-MAIL ADDRESS	PROJECT ROLE
Eileen Furey	Associate Regional Counsel	U.S. EPA, Region 5	312.886.7950	furey.eileen@epa.gov	Counsel for U.S. EPA
Jennifer Hale	Environmental Manager	Weyerhaeuser Company	253.924.3746	jennifer.hale@weyerhaeuser.com	Project Manager for Weyerhaeuser
Michael Berkoff	Remedial Project Manager	U.S. EPA, Region 5	312.353.8983	berkoff.michael@epa.gov	Remedial Project Manager for 12th Street Landfill
Sam Borries	On-Scene Coordinator	U.S. EPA, Region 5	312.353.8360	borries.samuel@epa.gov	On-Scene Coordinator for the TCRA
Jim Saric	Remedial Project Manager	U.S. EPA, Region 5	312.886.0992	saric.james@epa.gov	Remedial Project Manager for Kalamazoo River OU
Kathy Huibregtse	Principal-In-Charge	RMT, Inc.	262.879.1212	kathy.huibregtse@rmtinc.com	Senior Consultant - QA Manager

QAPP Worksheet #9-2 (*continued*)
Project Scoping Session Participants Sheet (June 6, 2007)

NAME	TITLE	AFFILIATION	TELEPHONE NUMBER	E-MAIL ADDRESS	PROJECT ROLE
Jim Hutchens (by Telephone)	Senior Project Manager	RMT, Inc.	262.879.1212	james.hutchens@rmtinc.com	RMT Project Manager for emergency actions in the former powerhouse discharge channel
Nathan Weber (By Telephone)	Project Engineer	RMT, Inc.	262.879.1212	Nathan.weber@rmtinc.com	Sampling Program Coordinator for emergency response actions in the former powerhouse discharge channel

Comments/Decisions:

- Discussed new project organization on remedial activities: Michael Berkoff – Landfills/Sam Chummar – Mills/ Jim Saric – River and oversight for all OUs. Communication distribution is to include all project managers, Sam Borries, and the MDEQ.
- Geotechnical information was reviewed to provide needed information to assess slope stability for the emergency action and additional insight to the U.S. EPA regarding impacts of tree removal on adjoining MDNR property. Overall, there are less soft paper residuals in the areas closest to the river, but soft material requires an adjustment to a 5:1 slope along the river face.
- Presented overall approach to the removal and relocation of former powerhouse discharge channel residuals to the top of the landfill, the regrading of the landfill slope, installation of 500-year flood erosion protection, installation at a hydraulic barrier through a clay plug, and the general configuration of the former powerhouse discharge channel activities and how they are integrated into the final RD/RA.
- Discussed and agreed to the option of a multi-area QAPP (desired) and the timeline for QAPP submittal.
- Discussed and agreed to intermediate submittals of portions of the design report since the schedule does not accommodate extended review in late July.
- Noted Weyerhaeuser's ongoing dialogue for ownership of the 12th Street Landfill property.

Action Items:

- Continue to prepare Quality Assurance Project Plan (QAPP) for target submittal date of June 21, 2007 - Weyerhaeuser
- Provide final bathymetry and visual sediment assessment scope document for the U.S. EPA's review by close-of-business June 6, 2007 - Weyerhaeuser
- Review and approve bathymetry and visual sediment assessment scope document by Friday, June 8, 2007 - U.S. EPA
- Continue to obtain engineering data and prepare design report - Weyerhaeuser

Consensus Decisions:

- Multi-Area QAPP is preferred in new format.
- Design report will be sent in sections for input or the schedule cannot be achieved.

QAPP Worksheet #9-3
Project Scoping Session Participants Sheet (December 7, 2007)

Project Name: 12th Street Landfill (Operable Unit No. 4) RD/RA

Projected Date(s) of Sampling: 2008 **Site Name:** Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

RMT Project Managers: Linda Hicken **Location of Scoping Session:** U.S. EPA Region 5, Chicago, Illinois

Date of Session: December 7, 2007

Scoping Session Purpose: To kick off the Remedial Design project

NAME	TITLE	AFFILIATION	TELEPHONE NUMBER	E-MAIL ADDRESS	PROJECT ROLE
Eileen Furey	Associate Regional Counsel	U.S. EPA, Region 5	312.886.7950	furey.eileen@epa.gov	Counsel for U.S. EPA
Michael Berkoff	Remedial Project Manager	U.S. EPA, Region 5	312.353.8983	berkoff.michael@epa.gov	Project Manager for 12th Street Landfill
Paul Bucholtz (by telephone)	Superfund Section - Specialized Sampling Unit - Project Manager	MDEQ	517.373.8174	bucholtp@michigan.gov	MDEQ Project Manager
Jennifer Hale	Environmental Manager	Weyerhaeuser Company	253.924.3746	jennifer.hale@weyerhaeuser.com	Project Coordinator for Weyerhaeuser
Joe Jackowski (by telephone)	Senior Legal Counsel	Weyerhaeuser Company	253.924.3461	joe.jackowski@weyerhaeuser.com	Counsel for Weyerhaeuser
Mark Schneider (by telephone)	Attorney at Law	Perkins Coie	206.359.8627	mwschneider@perkinscoie.com	Outside Counsel for Weyerhaeuser
Jeff Keiser	Project Manager	CH2M Hill	414.847.0382	jeff.keiser@ch2m.com	Oversight Contractor to the U.S. EPA
Linda Hicken	Senior Project Manager	RMT, Inc.	608.662.5307	linda.hicken@rmtinc.com	Project Manager for 12th Street Landfill RD/RA

QAPP Worksheet #9-3 (*continued*)
Project Scoping Session Participants Sheet (December 7, 2007)

NAME	TITLE	AFFILIATION	TELEPHONE NUMBER	E-MAIL ADDRESS	PROJECT ROLE
Jim Hutchens	Senior Project Manager	RMT, Inc.	262.879.1212	james.hutchens@rmtinc.com	Project Manager for Emergency Actions in the former powerhouse channel
Kathy Huibregtse	Vice President	RMT, Inc.	262.879.1212	kathy.huibregtse@rmtinc.com	Principal-In-Charge - QA Manager

QAPP Worksheet #9-3 (*continued*)
Project Scoping Session Participants Sheet (December 7, 2007)

Comments/Decisions:

- The meeting participants discussed lessons learned from the Emergency Action in the former powerhouse channel in 2007.
- The deadlines the 12th Street Landfill RD/RA have not been triggered. In the context of compliance with the SOW, the RD process was initiated with the submittal of the design of the erosion control measures along the eastern slope of the landfill as part of the Emergency Action in 2007. Weyerhaeuser recommended that the timeline for development of the RD Workplan be started on January 7, 2008.
- Weyerhaeuser summarized which components of the remedy were completed as part of the Emergency Action and which are to be completed as part of the Remedial Action. The U.S. EPA concurred.
- The potential need for a leachate collection system can be evaluated using existing information. Collection of additional field data is not anticipated.
- A gas extraction system will likely be installed with the landfill cover system. Limited field data may be collected as part of the predesign investigation.
- A schedule for accelerating the RD process should be presented in the RD Workplan.
- Weyerhaeuser presented a conceptual plan for an eco-park as a potential alternative approach for long-term site closure. The U.S. EPA advised engaging the natural resource trustees early on the idea.
- The findings pertaining to the 12th Street Landfill that were contained in the first 5-Year Review Report for the entire Kalamazoo River Superfund site were constrained by the options for evaluating the protectiveness of a remedy under the Superfund program. As necessary, the U.S. EPA will clarify for the public any potential misperceptions regarding the protectiveness of the remedy.
- The U.S. EPA is planning to host a site-wide discussion of the appropriate period of time in which wetland/floodplain areas may be inundated for purposes of determining clean up criteria for ecological receptors.

Action Items:

- Weyerhaeuser to provide more frequent updates to the U.S. EPA during periods of high activity in the field (e.g., daily e-mails as appropriate).
- As applicable, Weyerhaeuser to discuss options for field modifications with U.S. EPA's field representative.
- Weyerhaeuser to send a letter to the U.S. EPA, for inclusion in the Administrative Record, clarifying certain statements and conclusions in the first 5-Year Review Report.
- U.S. EPA to determine the appropriate mechanism for correcting the inaccurate statement in the first 5-Year Review Report that Weyerhaeuser needs to install a fence around the landfill by December 2007.
- U.S. EPA to provide a list of information needed to facilitate future decision making.
- U.S. EPA/MDEQ to obtain for Weyerhaeuser workplans/reports related to the management of landfill gas at the operable units.
- A meeting to discuss the preliminary scope of the predesign studies will be scheduled in early January 2008.

Consensus Decisions:

- A fence around the landfill is not required by December 2007, as stated in the first 5-Year Review Report. As required in the ROD, a perimeter fence is required as part of the RA.

QAPP Worksheet #9-4
Project Scoping Session Participants Sheet (January 9, 2008)

Project Name: 12th Street Landfill (Operable Unit No. 4) RD/RA

Projected Date(s) of Sampling: 2008 **Site Name:** Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

RMT Project Managers: Linda Hicken **Location of Scoping Session:** Conference Call

Date of Session: January 9, 2008

Scoping Session Purpose: To discuss the scope of the predesign studies

NAME	TITLE	AFFILIATION	TELEPHONE NUMBER	E-MAIL ADDRESS	PROJECT ROLE
Michael Berkoff	Remedial Project Manager	U.S. EPA, Region 5	312.353.8983	berkoff.michael@epa.gov	Project Manager for 12th Street Landfill
Paul Bucholtz	Superfund Section - Specialized Sampling Unit - Project Manager	MDEQ	517.373.8174	bucholtp@michigan.gov	MDEQ Project Manager
Jennifer Hale	Environmental Manager	Weyerhaeuser Company	253.924.3746	jennifer.hale@weyerhaeuser.com	Project Coordinator for Weyerhaeuser
Jeff Keiser	Project Manager	CH2M Hill	414.847.0382	jeff.keiser@ch2m.com	Oversight Contractor to the U.S. EPA
Linda Hicken	Senior Project Manager	RMT, Inc.	608.662.5307	linda.hicken@rmtinc.com	Project Manager for 12th Street Landfill RD/RA
Mike Amstadt	Senior Design Engineer	RMT, Inc.	608.662.5271	mike.amstadt@rmtinc.com	Design QA
Eric Watruba	Design Engineer	RMT, Inc.	608.662.5226	eric.watruba@rmtinc.com	Lead engineer
Kathy Huibregtse	Vice President	RMT, Inc.	262.879.1212	kathy.huibregtse@rmtinc.com	Principal-In-Charge - QA Manager

QAPP Worksheet #9-4 (*continued*)
Project Scoping Session Participants Sheet (December 7, 2007)

Comments/Decisions:

- The timeline for development of the RD Workplan started on January 7, 2008. Although the Consent Decree allows 60 days for submittal of the RD Workplan, in order to expedite the RD process, Weyerhaeuser plans to submit the RD Workplan on February 21, 2008.
- Weyerhaeuser presented a summary of the existing information collected during historical investigations at OU-4, and described the additional information to be collected to support the grading plan, to assess the extent of visible residuals in certain areas (the woodlands and asphalt plant property), and to assess constructability in the wetland.
- The potential need for a leachate collection system will be evaluated using existing information. No additional field data is needed.
- A passive gas collection system will be installed with the landfill cover system. Limited field data may be collected as part of the predesign investigation.
- A schedule for streamlining the RD deliverables and schedule will be presented in the RD Workplan.
- As applicable, Weyerhaeuser will identify potential scenarios under which field modifications (e.g., additional test pits or change in methods) may be needed.

Action Items:

- Weyerhaeuser plans to submit the RD Workplan on February 21, 2008.
- Weyerhaeuser will submit a Freedom of Information Act request to the MDEQ to facilitate its previous request for workplans/reports related to the management of landfill gas at the operable units.

Consensus Decisions:

- The Health and Safety Plan for the predesign field investigations will be submitted as part of the RD Workplan.

QAPP Worksheet #9-5
Project Scoping Session Participants Sheet (July and August, 2008)

Project Name: Plainwell Mill (Operable Unit No.7) RI/FS

Projected Date(s) of Sampling: 2008 **Site Name:** Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

RMT Project Managers: Jim Hutchens **Location of Scoping Session:** Conference Call

Date of Sessions: July 16, 2008, July 22, 2008, and August 28, 2008 via Conference Call

Scoping Session Purpose: To discuss the proposed Phase 2 of Addendum No. 1 and the Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment

NAME	TITLE	AFFILIATION	TELEPHONE NUMBER	E-MAIL ADDRESS	PROJECT ROLE
Sam Chummar	Remedial Project Manager	U.S. EPA, Region 5	312.886.1434	chummar.sam@epa.gov	Project Manager for Plainwell Mill
Paul Bucholtz	Superfund Section - Specialized Sampling Unit - Project Manager	MDEQ	517.373.8174	bucholtp@michigan.gov	MDEQ Project Manager
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Nathan Weber	Project Engineer	RMT, Inc.	262.879.1212	nathan.weber@rmtinc.com	Lead engineer
Kathy Huibregtse	Vice President	RMT, Inc.	262.879.1212	kathy.huibregtse@rmtinc.com	Principal-In-Charge - QA Manager

QAPP Worksheet #9-5 (*continued*)
Project Scoping Session Participants Sheet (July and August, 2008)

Comments/Decisions:

- Discussion and comment regarding the proposed analyte list
- Discussion and comment regarding the construction of the clay barrier configuration along the Plainwell Mill bank
- Coal Conveyance Tunnel
- Revised the Contingency Plan
- Discussion and comment regarding separating out the groundwater portion from the Addendum
- As applicable, Weyerhaeuser will identify potential scenarios under which field modifications (e.g., additional test pits, changes in location or methods) may be needed.

Action Items:

- Respond to Comments and Submit Revised Workplan – Completed (8/15/08)
- Conference Call on 8/27/08 to Discuss the Revisions (Outcomes listed in the consensus decisions)
- Submitted initial groundwater plan concepts to USEPA and MDEQ (8/20/08)

Consensus Decisions:

- Phase 2 of Addendum No. 1 will include sampling from pooled liquids in tests pits, but results will be for screening purposes only and will not be compared to Part 201 criteria
- Phase 2 of Addendum No. 1 will include excavation below the water table when conditions allow
- Characterization of material for offsite disposal will include a PID measurement
- Characterization of material for selected for disposal will be disposed of properly according to applicable state and federal regulations.
- Need to submit a full work plan for the Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment

QAPP Worksheet #10-1
Problem Definition - DQOs
(Emergency Response Activities in the Former Powerhouse Discharge Channel)

Step 1: State the problem.

In 2001, the United States Environmental Protection Agency (U.S. EPA) issued a Record of Decision (ROD) for the 6.5-acre 12th Street Landfill located in Otsego Township within Allegan County. The 2001 ROD consisted of 11 major components, including relocation of residuals present in the former powerhouse discharge channel to the landfill and construction of an erosion control system protective of a 500-year flood event. In February 2007, the U.S. EPA authorized a Time-Critical Removal Action (TCRA) to remove PCB-contaminated sediment in the former Plainwell Impoundment (a section of Operable Unit #5 of the Allied Paper/Portage Creek/Kalamazoo River Superfund Site). As part of the TCRA, the earthen section of the Plainwell Dam will be removed and the Kalamazoo River will be rerouted through the former powerhouse discharge channel. The 12th Street Landfill abuts the river and is located directly downstream of the earthen section of the Plainwell Dam.

Rerouting of the Kalamazoo River will impact paper residuals currently present in the channel and will create different erosion and flood conditions along the 12th Street Landfill riverbank. Weyerhaeuser Company (Weyerhaeuser) is planning to address these issues under Section XVII (Emergency Response), paragraph 67, of Weyerhaeuser's 2005 Consent Decree with the U.S. EPA. The emergency response activities will include removing residuals that are contiguous with the 12th Street Landfill, regrading the landfill slope adjoining the river, and constructing an erosion protection system consistent with the requirements of the ROD. Data collection activities during the emergency removal action will include surface water quality monitoring and effluent monitoring of treated dewatering fluids collected from the sediment containment area.

Various investigative activities have identified paper residuals along the bank of the 12th Street Landfill and within the former powerhouse discharge channel contiguous to the landfill. Additional data will be collected to assess residual PCB concentrations after remedial measures are completed.

Step 2: Identify the goal of the study.

The key goals of the post-removal action monitoring are as follows:

- Confirmation of the removal of visual wastewater residuals in the former powerhouse discharge channel that were contiguous with the 12th Street Landfill
- Documentation of post-removal topography along the Kalamazoo River

The key goals of monitoring during construction operations are as follows:

- Documentation of surface water quality during emergency actions
- Protection of water quality by application of a real-time in-field measurement of turbidity as an action level
- Protection of water quality by collection of confirmatory effluent samples from dewatering treatment operations

Step 3: Identify information inputs.

- Decision inputs for removal incorporate both the concentration and the distribution of PCBs in site media. Data to be collected to meet the goals of the removal action include a sufficient number of data points of acceptable quality to support any decisions.
- Decision input for water treatment is the comparison of upstream and downstream turbidity with an action level of 2 times the upstream turbidity.

QAPP Worksheet #10-1 (*continued*)
Problem Definition - DQOs
(Emergency Response Activities in the Former Powerhouse Discharge Channel)

Step 4: Define the boundaries of the sampling.

- The area considered as part of this action includes the 12th Street Landfill and the former powerhouse discharge channel located in Otsego Township, Allegan County (See Figure 1-1).

Step 5: Develop the analytical approach.

- Confirmation samples for PCBs in sediment will be analyzed on a quick-turn basis. Data will be used to adjust removal activities in the field as appropriate. The final decision on removal effectiveness will be based on validated confirmation results.
- Confirmation water quality samples will be tested for PCBs, phosphorus, and TSS using standard turnaround times. Data will be compared with turbidity samples to confirm or adjust action levels.

Step 6: Specify performance or acceptance criteria.

Visual observations and post-excavation sediment samples in the area(s) where residuals were removed will be used to confirm the horizontal and vertical extent of the excavation and the effectiveness of the removal action, as well as to determine whether additional sediment will be removed. Additional sampling will be performed if additional sediment is removed.

Step 7: Develop the plan for obtaining the data.

The overall QA objective is to develop and implement procedures for field sampling that will provide results to support the evaluation of site data consistent with NCP requirements. In general, spatially distributed grid sampling will be performed to provide representative distribution and an adequate number of samples The size of the grids and the number of sampling locations will depend upon the areal extent of sediment removal. Specific procedures for sampling, chain-of-custody, instrument calibration, analysis, reporting, and internal quality control are described in other worksheets of this QAPP.

QAPP Worksheet #10-2
Problem Definition - DQOs
(Emergency Response Activities on the Plainwell Mill Banks)

Step 1: State the problem.

In February 2007, the U.S. EPA authorized a Time-Critical Removal Action (TCRA) to remove PCB-contaminated sediment in the former Plainwell Impoundment (a section of Operable Unit 05 of the Allied Paper/Portage Creek/Kalamazoo River Superfund Site). As part of the TCRA, the earthen section of the Plainwell Dam will be removed and the Kalamazoo River will be rerouted through the former powerhouse discharge channel. Rerouting of the Kalamazoo River will impact paper residuals currently present in the channel and will create different erosion and flood conditions along the Plainwell Mill banks. Various investigative activities have identified paper residuals along the bank of the Plainwell Mill.

The actions being taken for the Plainwell Impoundment TCRA could cause a release of hazardous substances from the Plainwell Mill banks, so the U.S. EPA has authorized Weyerhaeuser to address the issue and thus prevent abate or minimize a release or potential release. Work will include excavating or containing paper residuals that are present in the floodplain and bank areas near the river along the former Plainwell Mill, reshaping of banks in those locations, and possible construction of erosion controls to minimize bank undercutting. These bank areas are part of the Kalamazoo River Operable Unit. Specific emergency response work to be performed by Weyerhaeuser in addressing the threatened release will be designed and performed in a manner that considers the implications of the City of Plainwell future land use concepts and is generally consistent with applicable provisions of the Settlement Agreement and the Former Plainwell Impoundment Time-Critical Removal Action Design Report.

Data collection activities during the emergency action will include surface water quality monitoring and effluent monitoring of treated dewatering fluids collected from the sediment containment area. Additional data will be collected to assess residual PCB concentrations after remedial measures are completed.

Step 2: Identify the goal of the study.

The key goals of the post Emergency Action monitoring are as follows:

- Confirmation of the removal of visual wastewater residuals on and immediately adjacent to the Plainwell Mill banks.
- Documentation of post Emergency topography along the Kalamazoo River.

The key goals of monitoring during construction operations are as follows:

- Documentation of surface water quality during emergency actions.
- Protection of water quality by application of a real-time in-field measurement of turbidity as an action level.
- Protection of water quality by collection of confirmatory effluent samples from dewatering treatment operations.

Step 3: Identify information inputs.

- Decision inputs for excavation incorporate both the concentration and the distribution of PCBs and the associated visual presence of paper residuals in floodplain soils, near shore sediments and limited bank soil. Data to be collected to meet the goals of the Emergency Action include a sufficient number of data points of acceptable quality to support any decisions.
- Decision input for water treatment is the comparison of upstream and downstream turbidity with an action level of 2 times the upstream turbidity.

Step 4: Define the boundaries of the sampling.

- The area considered as part of this action includes the Plainwell Mill Banks located in Gun Plain Township, Allegan County (see Figure 1-1).

QAPP Worksheet #10-2 (*continued*)
Problem Definition - DQOs
(Emergency Response Activities on the Plainwell Mill Banks)

Step 6: Specify performance or acceptance criteria.
Visual observations and post-excavation bank soil, floodplain soil and sediment samples in the area(s) where residuals were removed will be used to confirm the horizontal and vertical extent of the excavation and the effectiveness of the removal action, as well as to determine whether additional soil and sediment will be removed. Additional sampling will be performed if additional soil and sediment is removed.
Step 7: Develop the plan for obtaining the data.
The overall QA objective is to develop and implement procedures for field sampling that will provide results to support the evaluation of site data consistent with NCP requirements. In general, spatially distributed grid sampling will be performed to provide representative distribution and an adequate number of samples The size of the grids and the number of sampling locations will depend upon the aerial extent of banks soil, floodplain soil and sediment removal. Specific procedures for sampling, chain-of-custody, instrument calibration, analysis, reporting, and internal quality control are described in other worksheets of this QAPP.

QAPP Worksheet #10-3
Problem Definition - DQOs
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)

Step 1: State the problem.

Operable Unit #4 (OU-4) of the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site (Kalamazoo River Superfund Site) consists of the former 12th Street Landfill and the four areas outside the landfill where PCB-contaminated residual material has been observed. The 6.5 acre landfill is situated on an approximately 24-acre property with about 17 acres of wetlands. The additional portions of OU-4 that are located outside the landfill property include the woodland area owned by the State of Michigan (State) under the management of the Michigan Department of Natural Resources (MDNR), the asphalt plant operation adjacent to the landfill, and the former powerhouse discharge channel. Contamination in the former powerhouse discharge channel was addressed as part of Emergency Actions implemented in 2007.

Paper residuals from the former Plainwell Mill (mill), located in Plainwell, Michigan, were disposed in the 12th Street Landfill during the period from approximately 1955 to 1981. The landfill reportedly also accepted solid waste from the mill during part of its period of active operation. A number of investigations have been performed at the site. The investigations have confirmed the nature of the material in the landfill and have shown that paper residuals are present in certain areas outside of the landfill (i.e., in the wetlands to the north/northwest, the asphalt plant property, and the State property). Some of the residuals/native soil beyond the toe of the landfill (i.e., outside the footprint of the landfill) may contain PCBs at concentrations exceeding State of Michigan or Kalamazoo River Superfund Site-specific ecological risk-based criteria.

A Record of Decision (ROD) for OU-4 was issued on September 28, 2001. The major components of the selected remedy include the following: "(1) the excavation and relocation into the landfill of contaminated residuals currently in the woodland, wetlands, and adjacent property, and the residuals in the former powerhouse discharge channel that are contiguous with the eastern side of the landfill, followed by the construction of a containment system; (2) the excavation and relocation into the landfill of the east side of the landfill along the former powerhouse discharge channel to create a buffer zone sufficient to insure no hydraulic connection between the wastes within the landfill containment system and the Kalamazoo River or the former powerhouse discharge channel; (3) the restoration of areas excavated, cleared and grubbed, or otherwise affected by the remedial action; (4) the construction of a side wall containment system around the outside of the landfill; (5) the construction of a cover (cap) over the landfill; (6) the installation of an appropriate groundwater monitoring network and the performance of long-term groundwater monitoring; (7) the performance of short-term surface water monitoring; (8) the placement of deed restrictions; (9) the construction of a fence, permanent markers, and warning signs; (10) the investigation of the need for a leachate collection system and, if deemed necessary, the construction of a leachate collection system; and (11) the implementation of long-term maintenance and post-closure care."

In January 2005, Weyerhaeuser Company (Weyerhaeuser) negotiated a Consent Decree (CD) with the U.S. EPA (Civil Action No. 1:05-CV0003) that established the Remedial Design (RD) and Remedial Action (RA) activities for the 12th Street Landfill. This revision of the Multi-Area Quality Assurance Project Plan (QAPP) has been prepared in fulfillment of the requirements for an RD Workplan for the 12th Street Landfill that are contained in the CD and the Statement of Work (SOW).

The RD Workplan for the 12th Street Landfill includes a number of predesign investigations, some of which will involve field data collection and visual observations. Field data collection activities will include visual information obtained by the advancement of test pits, visual information obtained by the advancement of Geoprobe® borings, and gas concentration measurements (methane, carbon dioxide, and oxygen) in existing monitoring wells and in the Geoprobe® borings advanced as part of the predesign investigation.

The field investigations will involve (1) a determination of the extent of visible paper residuals beyond the landfill footprint, (2) the collection of data to support the grading design for the final landfill cover, and (3) the collection of data for use in the design of a landfill gas venting system. Sufficient information is available from previous investigations to evaluate the potential need for a leachate collection system. No additional field information is needed to support this objective.

QAPP Worksheet #10-3 (*continued*)
Problem Definition - DQOs
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)

Step 2: Identify the goal of the study.

The key goals of the data collection activities for the predesign investigations are as follows:

Extent of Paper Residuals Beyond Landfill Footprint

Wetland

- To confirm the approximate areal extent of visible paper residuals beyond the toe of the landfill as delineated based on the results from previous investigations.
- To assess the degree of difficulty in distinguishing the visible paper residuals from the native soil.
- To evaluate potential constructibility issues associated with working in the wetland north of the landfill.

Asphalt Plant/State Properties

- To delineate the areal extent and to better estimate the depth of visible paper residuals on the asphalt plant property to the southwest and on the State property to the southeast, in order to support discussions with owners of these adjacent properties concerning access for conducting the remedial actions on the properties required by the ROD.

Grading Design

- To better estimate the depth of the paper residuals along the property boundaries with 12th Street, the asphalt plant to the southwest, and with the State property to the southeast, in order to reduce uncertainties in designing the final landfill grades.

Landfill Gas Management

- To collect readily accessible information about the subsurface landfill gas conditions at the 12th Street Landfill that may be useful in designing a passive gas venting system for the landfill.

QAPP Worksheet #10-3 (*continued*)
Problem Definition - DQOs
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)

Step 3: Identify information inputs.

Extent of Paper Residuals Beyond Landfill Footprint

Wetland

- Evaluating constructibility issues – Information inputs will include visual observations of the depth to groundwater in the wetland and any related complications that are encountered during excavation of the near-surface soil.
- Confirming areal extent and distinguishing the visible paper residuals from the native soil – Information inputs will include visual observations of paper residuals being present in the soil beyond the toe of the landfill.

Asphalt Plant/State Properties

- Delineating areal extent and better estimating the depth of visible paper residuals – Information inputs will include visual observations of paper residuals being present in the soil on these properties.

Grading Design

- Better estimating the depth of the paper residuals along the property boundaries with 12th Street, the asphalt plant to the southwest, and with the State property to the southeast - Information inputs will include visual observations of paper residuals being present in these areas and classification of the materials encountered based on the procedures outlined in ASTM D2488 (i.e., soil types).

Landfill Gas Management

- Evaluating subsurface landfill gas concentrations– Information inputs will include measurements of concentrations of methane, carbon dioxide, and oxygen in the vadose zone at the site.

The information that is collected for determining the extent of paper residuals beyond the landfill footprint and for the grading design will be supplemented with similar visual information recorded during previous investigations at the 12th Street Landfill site (see Worksheet #13-1). No soil gas measurements have been collected to-date.

Step 4: Define the boundaries of the sampling.

- The area considered as part of this action includes the 12th Street Landfill property, and adjacent asphalt plant and State properties, located in Otsego Township, Allegan County (see Figure 1-1).

Step 5: Develop the analytical approach.

- No samples will be collected for laboratory analytical testing. Gas sampling will be performed on-site using a portable combustible gas meter.

QAPP Worksheet #10-3 (*continued*)
Problem Definition - DQOs
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)

Step 6: Specify performance or acceptance criteria.

The acceptance criteria for the predesign data collection activities are having collected sufficient information to meet the study objectives described under Step 2 above and sufficient information to proceed with the Remedial Design. If these criteria are not met, additional data will be collected in a similar manner to meet these objectives.

Step 7: Develop the plan for obtaining the data.

Extent of Paper Residuals Beyond Landfill Footprint

Wetland

- Evaluating constructibility issues and confirming areal extent and distinguishing the visible paper residuals from the native soil – Advance approximately 3 test pits

Asphalt Plant/State Properties

- Delineating areal extent and better estimating the depth of visible paper residuals – Advance approximately 3 test pits within the asphalt plant property and approximately 4 test pits within the State property

Note: In the event that in-field conditions limit the use of test pit excavating equipment (e.g., a backhoe), other tools, such as hand augers or shovels, may be used instead. In such instances, the U.S. EPA project manager, or designated alternate, will be contacted to discuss the situation and to agree on a course of action. In addition, Weyerhaeuser will conduct a kick-off meeting with the U.S. EPA and its field representative(s) at the outset of the predesign field investigation. During this meeting, the schedule for conducting the predesign field activities and the process for obtaining U.S. EPA approval of field modifications will be reviewed.

Grading Design

- Better estimating the depth of the paper residuals along the property boundaries with 12th Street, the asphalt plant to the southwest, and with the State property to the southeast – Advance approximately nine Geoprobe® borings into the 12th Street Landfill at select locations where fill material is believed to extend beyond the property boundary to the southwest and to the southeast.
- Advance a minimum of two soil borings (RDB-10 and RDB-11) near the southern end of the landfill, as shown on Figure 2-3, to confirm the thickness of the fill in this area. Advance the borings approximately 5 feet into the native soil underlying the fill or to refusal. The locations of these borings may be adjusted in the field as necessary to avoid underground or aboveground utility lines. Additional borings may be installed to the north of the initial borings as may be deemed useful by Weyerhaeuser, in consultation with oversight agencies as needed, for purposes of designing the landfill cover (e.g., if fill material is not encountered at a location where existing data that indicate fill is present).

Landfill Gas Management

- Evaluating subsurface landfill gas concentrations – As readily accessible, measure gas concentrations (methane, oxygen, and carbon dioxide) and pressures (vacuum) at the existing groundwater monitoring wells screened in the vadose zone (MW-6A, MW-7A, and MW-8A). In addition, measure gas concentrations at each Geoprobe® boring used to better estimate the depth of the paper residuals along the property boundaries to the southwest and to the southeast. Pressures that may have developed within the groundwater monitoring wells caused by excess landfill gas (if present) will also be measured. A passive gas venting system can be designed without the above information. If these data cannot be readily obtained, additional efforts will not be employed to collect the information.

QAPP Worksheet #10-4
Problem Definition - DQOs
(Phase 2 of Addendum No. 1 – Test Pits and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)

Step 1: State the problem.

Phase 2 of Addendum No. 1 – Test Pits

The Plainwell Mill has been identified as Operable Unit #7 (OU-7) of the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site (Kalamazoo River Superfund Site). The Addendum activities are being conducted in response to discovery of elevated concentrations of polychlorinated biphenyls (PCBs) and oil impacted soil along the Plainwell Mill banks during the separate Plainwell Mill Banks Emergency Response activities. These discoveries prompted several responses including a USEPA letter to Weyerhaeuser dated February 19th, 2008 that requested additional information to be gathered along the Mill banks to determine the following:

- The cause of the oily sheen observed during the late January excavation, including consideration of whether a non-aqueous phase liquid (NAPL) was the cause;
- Whether NAPL is the cause of high PCB concentration at sample locations: PPM-SD-041, PEX-1, and PEX-2; and
- Whether NAPL, if present, could be entering into the Kalamazoo River.

Collecting information to answer the above questions is being completed in a phased manner. The initial phase consisted of a detailed review of available historical and Phase I and II Environmental Site Assessments results supplemented by a Geophysical survey between the Mill buildings and Kalamazoo River. As a result of the initial phase of activities a preliminary site conceptual model was found identifying several targets for additional investigation during the Phase 2 field activities.

The Phase 2 Work Plan for Addendum No. 1 at the Plainwell Mill has been prepared to identify sampling locations, methods, and the proposed analytical plan for the targeted areas. In general, test pits are proposed to assess the validity of the preliminary site conceptual model and additional test pits will be added if needed during the field investigation to identify any additional conduits or confirm the nature, and extent of contamination. The analytical program will consist of the primary issues observed during the Emergency Action, PCBs and TPH (DRO/GRO), with supplemental sampling for RCRA metals, PAHs, and VOCs.

Plainwell Mill RI/FS – Phase I Groundwater Assessment

The Plainwell Mill has been identified as Operable Unit #7 (OU-7) of the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site (Kalamazoo River Superfund Site). The groundwater reported to date suggest that the site operations had limited adverse impact on groundwater quality. However, since the groundwater samples previously collected on the site were taken from temporary wells and were not collected under a QAPP, the groundwater quality is uncertain for the purposes of risk assessment and comparison to ARARS. In response to these and ongoing concerns regarding the potential for unknown releases a limited groundwater well network is proposed for the Plainwell Mill. The proposed wells will target areas of interest including:

- Mill Buildings,
- Coal Conveyance Tunnel,
- Former Transformer Pad,
- Oil Impacted Soil,
- No. 6 Fuel Oil AST,
- Areas of Elevated PCB Concentrations
- Former Sludge Dewatering Lagoons

A total of 12 groundwater wells are expected to be installed to target the above areas. The analytical program will consist of PCBs, PAHs, RCRA metals, and VOCs. In addition, to providing analytical data the groundwater wells will also provide information regarding the interface between the groundwater system and surface water, the direction of the groundwater gradient, the relationship between fill elevations and the surface water elevation, and general subsurface conditions at the site.

QAPP Worksheet #10-4 (*continued*)
Problem Definition - DQOs
(Phase 2 of Addendum No.1 – Test Pits and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)

Step 2: Identify the goal of the study.

The key goals of the data collection activities for Phase 2 of Addendum No.1 and the Plainwell Mill RI/FS – Phase I Groundwater Assessment are as follows:

Phase 2 of Addendum No. 1 – Test Pit Investigation

- **Identify Source**: Identify, as much as possible, a likely cause of the oily sheen observed near the former transformer pad, with focus on the possible presence of NAPL. To assess the degree of difficulty in distinguishing the visible paper residuals from the native soil.
- **Determine Relationships**: Determine if there is a relationship between the oil impacted soil near the transformer pad and the elevated PCB concentrations found at PM-SD-041, PEX-1, and PEX-2
- **Assess NAPL**: Determine whether NAPL, if present, is entering the Kalamazoo River
- **Define Nature of Contamination**: Determine if the oil impacted soil is present in soils and fill near the transformer pad and at locations PM-S-041, PEX-1, and PEX-2 contain petroleum products and/or PCBs.
- **Develop Next Steps**: Determine immediate actions needed and how the next phase of investigation should be integrated with other RI activities.

Plainwell Mill RI/FS – Phase I Groundwater Assessment

- Understanding the interface of the groundwater system with the River system
- Determining the direction of the groundwater gradient
- Understand the relationship between the site residual and/or fill elevations and the water table
- Obtain information regarding the groundwater quality at the facility
- Provide an initial assessment of the fuel oil lines exterior to the coal tunnel additional observations

QAPP Worksheet #10-4 (*continued*)
Problem Definition - DQOs
(Phase 2 of Addendum No.1 – Test Pits and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)

Step 3: Identify information inputs.

Phase 2 of Addendum No. 1 – Test Pits

- Define Extent of Oil Impacted Soil – Information inputs will include approximately 10 test pits in and near the site of the previously identified oil impact soil to visually observe soil types, location of petroleum impacts, and depth to groundwater. In addition, results from sample analysis for soil PCB and total petroleum hydrocarbons (TPH) (gasoline and diesel range) concentrations in each of the test pits.
- Define Extent of Elevated PCB Concentrations – Information inputs will include approximately 5 test pits near the elevated PCB concentrations to visually observe the soil types, location of petroleum impacts, and depth to groundwater. Currently the relationship between visual cues and PCB concentrations is not well understood making the sample analysis for PCBs and DRO/GRO a critical input to the process.
- Additional supplemental analysis for RCRA metals, VOCS, and PAHs may also be useful to further define other potential contamination at the site.
- Verify Site Geophysical Survey – The proposed test pits will also provide verification of soil types and assist in identifying other subsurface features.
- Free Product Characterization – If free product is observed in any of the test pits it may be useful to collect a sample to determine specific gravity and viscosity.
- Pooled water may be sampled for analysis, but will be collected for screening purposes only.

Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment

- Evaluate Site Groundwater Impacts – Information inputs will include a groundwater monitoring network of 12 wells at the site. Groundwater will be sampled for PCBs, VOCs, RCRA metals, and PAHs.
- Evaluate Groundwater Flow Direction – The installation of a site wide network (12 wells) and three additional staff gages will assist in understanding the subsurface flow conditions at the site.
- General Subsurface Information – The installation of the wells will also provide information regarding the soil types and any fill present at the location of each individual well.

Step 4: Define the boundaries of the sampling.

Phase 2 of Addendum No. 1 – Test Pits

- The area considered as part of this action includes the former Plainwell Mill Bank near the Mill Buildings, located in Plainwell, Michigan.

Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment

- The area considered as part of this action includes the former Plainwell Mill Property, located in Plainwell, Michigan.

QAPP Worksheet #10-4 (*continued*)
Problem Definition - DQOs
(Phase 2 of Addendum No.1 – Test Pits and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)

Step 5: Develop the analytical approach.

Phase 2 of Addendum No. 1 – Test Pits

- The primary targeted analytical data to be collected from the test pits is soil samples for analysis of PCBs, and TPH (DRO/GRO). Supplemental soil samples may be collected for analysis of VOCs, RCRA metals, and PAHs.
- Test pit installation and soil sampling techniques will follow SOPs 7 and 8, respectively.
- Onsite field screening using visual cues, odors, and PID equipment (SOP F-6) will be used to assist in selecting samples for analysis and refinement of test pit locations.

Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment

- The primary targeted analytical data to be collected from the groundwater wells is groundwater samples for analysis of PCBs, RCRA metals, and PAHs.
- A flow-through cell will also be used (SOP F-10) for sampling groundwater for turbidity, pH, conductivity, and dissolved oxygen.
- Staff gages will be installed to collect river water elevations in concert with groundwater well elevations (SOP F-11).

Step 6: Specify performance or acceptance criteria.

Phase 2 of Addendum No. 1 (Test Pits)

- The acceptance criteria for this activity is having collected sufficient data and observations to meet the objectives identified in Step 2 above and allow sufficient information to continue refinement of the site conceptual model. In addition, the collected data must meet the appropriate analytical QA/QC requirements to be considered valid (Worksheet 12, Worksheet 28, and Attachment 1-1).

Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment

- The acceptance criteria for this activity is having collected sufficient data and observations to meet the objectives identified in Step 2 above and allow sufficient information to continue refinement of the site conceptual model. In addition, the collected data must meet the appropriate analytical QA/QC requirements to be considered valid (Worksheet 12, Worksheet 28, and Attachment 1-1).

QAPP Worksheet #10-4 (*continued*)
Problem Definition - DQOs
(Phase 2 of Addendum No.1 – Test Pits and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)

Step 7: Develop the plan for obtaining the data.

Phase 2 of Addendum No. 1 (Test Pits)

Due to the nature of the investigative areas likely to contain fill material and or construction debris, test pits have been selected as the best method for successful site characterizations. Test pits will be advanced to either native soils or the water table using an excavator, taking care to minimize site disturbances to the extent practical. The test pits will be placed along two areas between the former Mill buildings and the Kalamazoo River banks (the oil impacted soil and elevated PCB areas).

Approximately 10 test pits will be located within the site of the oil impacted soil. These test pits will attempt to identify the source and the extent of the oil impacted soil and specific test pit locations will be adjusted depending upon field observations. An additional five test pits will be placed in the areas where elevated PCBs were detected (near possible fill material). The need for additional test pits will be determined based upon field observations and discussion with the USEPA project manager or designated representative.

Soil samples collected from each test pit will be analyzed for PCBs and total petroleum hydrocarbons (GRO/DRO). Samples will also be visually classified for soil types and screened with a photoionization detector. Up to five selected samples will be submitted for analysis of RCRA metals, PAHs, and VOCs. If free product is observed in any of the test pits a sample will be collected for general physical characterization including specific gravity and viscosity.

Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment

Based upon prior site usage and previous investigative activities at the former Plainwell Mill site, the investigation areas were divided into three areas of concern: 1) the mill building area, 2) the northcentral portion of the site, and 3) the former wastewater sludge dewatering area. Data gaps from the prior investigations were discussed in the draft Plainwell Mill RI/FS Work Plan and the basis for each well location are included in the RI/FS Work Plan for the Initial Groundwater Investigation.

The initial phase of groundwater monitoring involves installing 12 constructed groundwater table monitoring wells to assess the shallow groundwater system. Monitoring wells will be constructed of 2-inch diameter PVC pipe placed into a 6 to 8-inch diameter drilled well annulus according to procedures described in the attached Proposed Monitoring Well Installation Procedures. A 10-foot long well screen will be located to intersect the water table based upon an initial assessment of the variability of the river stage elevation and the depth to groundwater. Since the surface water elevation can vary by several feet depending upon rainfall events, the groundwater elevation will reflect an unknown degree of variation. The longer well screen provides more certainty that the screen will intersect the water table over a wider range of site conditions.

Groundwater sampled from each location will be analyzed for volatile organic compounds (VOCs), polycyclic aromatic hydrocarbons (PAHs), RCRA metals, and polychlorinated biphenyls (PCBs). As a frame of reference, concentrations of measured constituents will be compared to applicable Michigan standards and be used to identifying possible upgradient areas that may require further investigation.

QAPP Worksheet #11-1
Project Quality Objectives/Systematic Planning Process Statements
(Emergency Response Activities in the Former Powerhouse Discharge Channel)

Who will use the data?

Weyerhaeuser, Inc.; RMT Inc.; and the overseeing agencies (the United States Environmental Protection Agency [U.S. EPA] and the Michigan Department of Environmental Quality [MDEQ]) will use the data.

What will the data be used for?

Surface Water Quality Monitoring:

The water quality monitoring data will be used to assess compliance with Applicable Relevant and Appropriate Requirements (ARARs) and to ensure that river water quality is not negatively impacted by the remedial actions in the former powerhouse discharge channel.

Sediment Sampling:

Sediment sampling data will be used to document the removal of residuals from the former powerhouse discharge channel that are contiguous with the 12th Street Landfill, including the effectiveness of the removal action and the presence of residual concentrations in the sediment.

Water Treatment System Monitoring:

The water treatment system monitoring data will be used to assess compliance with the discharge limits and other ARARs, and to ensure proper operation of the treatment system.

What types of data are needed (target analytes, analytical groups, field-screening, on-site analytical or off-site laboratory techniques, sampling techniques)?

Sampling and analytical protocols are defined later in this document.

Surface Water Quality Monitoring:

Water quality will be assessed by monitoring for turbidity and total PCBs. Turbidity samples will be taken to provide data instantaneously. Water samples will be submitted and sent to an off-site laboratory for total PCB analysis using EPA Method 608.

Sediment Sampling:

Field-screening will rely on the presence of visible residuals. Confirmatory sediment sampling will be assessed by sampling for total PCBs using off-site analytical testing.

Water Treatment Monitoring:

Data collected for the water treatment system will include total PCBs, total suspended solids (TSS), and total phosphorus as P. Total PCBs will be analyzed using EPA Method 608, TSS will be analyzed using EPA Method 160.2, and phosphorus will be analyzed using EPA Method 365.3. All samples will be analyzed by an off-site laboratory.

QAPP Worksheet #11-1 (*continued*)
Project Quality Objectives/Systematic Planning Process Statements
(Emergency Response Activities in the Former Powerhouse Discharge Channel)

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

Turbidity data must be collected with a meter that has a precision of at least + 1 nephelometric turbidity units (NTUs); all data should be collected with a similar meter, calibrated per the manufacturer's requirements.

PCB, TSS, and phosphorus data will be collected and analyzed in accordance with the performance standards of EPA Methods 608, 160.2, and 365.3, respectively.

Verifiable data are necessary to determine the need for additional sediment removal.

How much data are needed (number of samples for each analytical group, matrix, and concentration)?

How much data are needed will be determined based on the construction period and the extent of excavation.

QAPP Worksheet #11-1 (*continued*)
Project Quality Objectives/Systematic Planning Process Statements
(Emergency Response Activities in the Former Powerhouse Discharge Channel)

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

Surface Water Quality Monitoring:

- Turbidity will be taken prior to and following equipment placement in the work area; at the beginning of the work day, approximately hourly while work is performed; and at the end of the work day. The upstream turbidity samples will be taken at a location near the Plainwell spillway and then at two downstream locations approximately 200 feet and 300 feet downstream of the work area along the path of the water flowing past the work area. The samples will be collected from either a boat or a shore-mounted semipermanent pier. The compliance point is the sample located 300 feet downstream of the work area. The sample collected at 200 feet downstream will serve as a warning to address site actions if elevated results are measured.
- Turbidity of the water discharged to the river during dewatering will be monitored hourly at the pump discharge when dewatering operations are under way.
- River water grab samples will be taken for PCB analysis weekly while work is performed from one location upstream and two locations downstream of the work area (same as the aforementioned upstream and 200 and 300 feet downstream locations for turbidity).

Sediment Sampling:

- Samples will be collected as soon as practicable after completion of the former powerhouse discharge channel removal action.
- The channel area will be split into approximately 20 grids per 8,000-square foot area (*i.e.*, approximately 400 square feet per grid). Five grids will be selected from the approximately 20 grids in a semi-random fashion in order to ensure adequate coverage, and one sample will be collected from each selected grid (*i.e.*, a total of approximately five sediment samples per 8,000-square foot area).
- Specific sampling methods will depend upon the depth of the overlying water. If limited water is present, samples will be collected (*e.g.*, using a trowel) by targeting a sediment depth of 0-6 inches. If greater water depths are present, sediment samples will be collected with a Ponar-type grab sampler, a core sampler, or an alternative method suitable to the sediment conditions.

Water Treatment System Monitoring:

- Grab samples for PCB analysis will be taken twice per week from the effluent of the water treatment system.
- Grab samples for TSS will be taken twice per week from the effluent of the water treatment system.
- Grab samples for phosphorus will be taken twice per week from the effluent of the water treatment system.

Who will collect and generate the data?

RMT will collect the samples; the laboratory (WATS) will analyze the samples and issue data reports; and RMT will validate the data.

QAPP Worksheet #11-1 (*continued*)
Project Quality Objectives/Systematic Planning Process Statements
(Emergency Response Activities in the Former Powerhouse Discharge Channel)

How will the data be reported?

All data from surface water quality monitoring will be available immediately for alteration of field activities if results indicate unacceptable increases in turbidity. All turbidity results will be compiled and presented in the emergency action documentation report. Sediment sampling data will be reported as soon as available to confirm completion of the excavation, and validated data will be presented in the final summary report. Water treatment system monitoring data will be used as soon as they are available to verify that the treatment system is adequate and operating correctly, and validated data will be presented in the final summary report.

How will the data be archived?

Validated data will be archived in an appropriate database to ensure that information is readily available, searchable, and compatible with a GIS system.

QAPP Worksheet #11-2
Project Quality Objectives/Systematic Planning Process Statements
(Emergency Response Activities on the Plainwell Mill Banks)

Who will use the data?

Weyerhaeuser, RMT, and the overseeing agencies (U.S. EPA and the Michigan Department of Environmental Quality [MDEQ]) will use the data.

What will the data be used for?

Surface Water Quality Monitoring:

The water quality monitoring data will be used to assess compliance with Applicable Relevant and Appropriate Requirements (ARARs) and to ensure that river water quality is not negatively impacted by the remedial actions on the Plainwell Mill banks.

Soil and Sediment Sampling:

Soil and sediment sampling data will be used to document the excavation of residuals from the Plainwell Mill banks, including the effectiveness of the emergency action and the presence of residual concentrations in the soil and sediment.

Water Treatment System Monitoring:

The water treatment system monitoring data will be used to assess compliance with the discharge limits and other ARARs, and to ensure proper operation of the treatment system.

What types of data are needed (target analytes, analytical groups, field-screening, on-site analytical or off-site laboratory techniques, sampling techniques)?

Sampling and analytical protocols are defined later in this document.

Surface Water Quality Monitoring:

Water quality will be assessed by monitoring for turbidity and total PCBs. Turbidity samples will be taken to provide data instantaneously. Water samples will be submitted and sent to an off-site laboratory for total PCB analysis using EPA Method 608.

Soil and Sediment Sampling:

Field-screening will rely on the presence of visible residuals. Confirmatory soil and sediment sampling will be assessed by sampling for total PCBs using off-site analytical testing.

Water Treatment Monitoring:

Data collected for the water treatment system will include total PCBs, total suspended solids (TSS), and total phosphorus as P. Total PCBs will be analyzed using EPA Method 608, TSS will be analyzed using EPA Method 160.2, and phosphorus will be analyzed using EPA Method 365.3. All samples will be analyzed by an off-site laboratory.

QAPP Worksheet #11-2 (*continued*)
Project Quality Objectives/Systematic Planning Process Statements
Emergency Response Activities on the Plainwell Mill Banks

How "good" do the data need to be in order to support the environmental decision?
Turbidity data must be collected with a meter that has a precision of at least + 1 nephelometric turbidity units (NTUs); all data should be collected with a similar meter, calibrated per the manufacturer's requirements. PCB, TSS, and phosphorus data will be collected and analyzed in accordance with the performance standards of EPA Methods 608, 160.2, and 365.3, respectively. Verifiable data are necessary to determine the need for additional soil and sediment excavation.
How much data are needed (number of samples for each analytical group, matrix, and concentration)?
How much data are needed will be determined based on the construction period and the extent of excavation.

QAPP Worksheet #11-3
Project Quality Objectives/Systematic Planning Process Statements
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)

Who will use the data? Weyerhaeuser, RMT, and the overseeing agencies (U.S. EPA and the Michigan Department of Environmental Quality [MDEQ]) will use the data.
What will the data be used for? The data will be used for the design of certain components of the remedies required in the Record of Decision (ROD) for the 12th Street Landfill site, including: (1) the excavation and relocation of residuals into the landfill, (2) the installation of an engineered cover, and (3) the installation of a sidewall containment system. Specifically, the predesign investigation data, which is collected by the installation of test pits and Geoprobe® borings, will be used to confirm the extent of paper residuals beyond the landfill footprint and for the site grading design. In addition, subsurface landfill gas concentration data will be obtained for use in the design of a passive gas venting system.
What types of data are needed (target analytes, analytical groups, field-screening, on-site analytical or off-site laboratory techniques, sampling techniques)? ***Extent of Paper Residuals Beyond Landfill Footprint –*** On-site visual observations obtained by the installation of test pits ***Grading Design –*** On-site visual observations obtained by the installation of Geoprobe® borings and the classification of soil types according to ASTM D2488 ***Landfill Gas Management –*** On-site field measurements of methane, carbon dioxide, and oxygen in the vadose zone at the site, using a portable combustible gas meter, and pressure, using a pressure gauge.

QAPP Worksheet #11-3
Project Quality Objectives/Systematic Planning Process Statements
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)

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

In order to meet the objectives of the pre-design investigations, the data collection activities must achieve the following:

Extent of Paper Residuals Beyond Landfill Footprint – To determine the thickness of the paper residuals in select locations, test pits must be excavated to a depth sufficient to reach the native soil underlying the paper residuals. If visible residuals are observed in the end of a test put furthest from the landfill, the test pit may be extended further away from the landfill and/or an additional test pit (or more) may be excavated nearby in order to meet investigative objectives. If an additional test pit (or more) is needed, the U.S. EPA project manager, or designated alternate, will be contacted to discuss the situation and to agree on a course of action. In the event that in-field conditions limit the use of test pit excavating equipment (e.g., a backhoe), other tools, such as hand augers or shovels, may be used instead. In such instances, the U.S. EPA project manager, or designated alternate, will be contacted to discuss the situation and to agree on a course of action. In addition, Weyerhaeuser will conduct a kick-off meeting with the U.S. EPA and its field representative(s) at the outset of the predesign field investigation. During this meeting, the schedule for conducting the predesign field activities and the process for obtaining U.S. EPA approval of field modifications will be reviewed.

Grading Design – To determine the thickness of the paper residuals, Geoprobe® borings must be installed to a depth sufficient to reach the native soil underlying the paper residuals.

Landfill Gas Management – A passive gas venting system will be incorporated into the landfill final cover regardless of the information that may be obtained from the predesign investigation. Additional information may be collected, if it is readily accessible, to supplement existing information/assumptions. To accurately determine the concentrations of methane, carbon dioxide, and oxygen in the vadose zone soil, the portable gas meter must be properly calibrated, and the readings on the gas meter must be allowed to stabilize before measurements are recorded. In addition, gas sampling ports must be properly installed on the existing monitoring wells (MW-6A, MW-7A, and MW-8A) and Post-Run Tubing (PRT) adapters must be properly installed on the landfill gas tubing that will be inserted into the Geoprobe® borings. SOP F-7 in the Field Sampling Plan provides detailed specifications for the equipment and procedures that will be used to complete these tasks.

How much data are needed (number of samples for each analytical group, matrix, and concentration)?

Extent of Paper Residuals Beyond Landfill Footprint – 3 test pits in the wetlands north of the landfill, 3 test pits on the asphalt plant property, and 4 test pits on the State property. In the event that in-field conditions limit the use of test pit excavating equipment (e.g., a backhoe), other tools, such as hand augers or shovels, may be used instead. In such instances, the U.S. EPA project manager, or designated alternate, will be contacted to discuss the situation and to agree on a course of action.

Grading Design – 9 Geoprobe® borings into the 12th Street Landfill; a minimum of 2 borings near the southern end of the landfill

Landfill Gas Management – A passive gas venting system will be incorporated into the final landfill cover regardless of the information that may be obtained from the predesign investigation. As readily accessible, gas measurements may be obtained from up to 3 existing groundwater monitoring wells screened in the vadose zone (MW-6A, MW-7A, and MW-8A) and in Geoprobe® borings installed as part of this investigation. As readily accessible, pressure will also be measured in up to 3 existing groundwater monitoring wells.

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

The data will be collected at the locations shown on Figures 2-2 and 2-3. The data collection activities are scheduled to be performed in May-June 2008, weather permitting. Sampling procedures are detailed in the Standard Operating Procedures (SOPs), which are included in the Multi-Area Field Sampling Plan, Revision 02.

QAPP Worksheet #11-3
Project Quality Objectives/Systematic Planning Process Statements
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)

Who will collect and generate the data?

RMT will collect the samples.

How will the data be reported?

Data from the predesign investigations will be presented in a section of the remedial design (RD) report or in a technical memorandum.

How will the data be archived?

Data will be archived in an appropriate database to ensure that information is readily available, searchable, and compatible with a GIS system.

QAPP Worksheet #11-4
Project Quality Objectives/Systematic Planning Process Statements
(Phase 2 of Addendum No. 1 - Test Pits & Plainwell Mill RI/FS – Phase I Groundwater Assessment)

Who will use the data?

Weyerhaeuser, RMT, and the overseeing agencies (U.S. EPA and the Michigan Department of Environmental Quality [MDEQ]) will use the data.

What will the data be used for?

Phase 2 of Addendum No. 1 - Test Pits

The data will be used in a Phased manner to investigate the nature and extent of contamination along the banks of the Plainwell Mill. Specifically, the data collected will be used to verify or deny the presence of free product, its source, and to identify the extent of the elevated PCB concentrations in excess of 4 mg/kg (MDEQ Part 201 Requirements).

Plainwell Mill RI/FS – Phase I Groundwater Assessment

The data associated with the groundwater assessment will be compared to applicable Part 201 criteria for RCRA metals, PCBs, PAHs, and VOCs. In addition, elevated concentrations in the groundwater will assist in identifying potential upgradient source areas that may require further investigation. After completion of problem identification, the data and future data collected at the site will be used to develop a site specific risk assessment and develop remedial options as part of a feasibility study to remediate excess risks.

What types of data are needed (target analytes, analytical groups, field-screening, on-site analytical or off-site laboratory techniques, sampling techniques)?

Phase 2 of Addendum No. 1 - Test Pits

*Extent of Oil Impacted Soil and if Free Product is Present***–** On-site visual observations obtained by the installation of test pits. Field screening to include observations for visible residuals, free product, petroleum impacts, PID readings, odors, and soil stratigraphy.

Targeted Analytes **–** Primarily analytes include PCBs and TPH (DRO/GRO) in soil. An additional subset of samples will be collected for RCRA metals, VOCs, and PAHs in soil. Soil samples will be collected using SOP F-1. If free product is observed a sample may be collected and submitted for general characterization such as specific gravity and viscosity. Laboratory techniques and SOPs are provided on Worksheet-19 and Worksheet-23.

Plainwell Mill RI/FS – Phase I Groundwater Assessment

Targeted Analytes **–** Primarily analytes include RCRA metals, PCBs, PAHs, and VOCs in groundwater. Groundwater well sampling procedures are provided in the Field and Sampling Plan. Laboratory techniques and SOPs are provided on Worksheet-19 and Worksheet-23.

QAPP Worksheet #11-4
Project Quality Objectives/Systematic Planning Process Statements
(Phase 2 of Addendum No. 1 - Test Pits & Plainwell Mill RI/FS – Phase I Groundwater Assessment)

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

In order to meet the objectives of the investigation activities, the data collection activities must achieve the following:

Phase 2 of Addendum No. 1 (Test Pits) – Due to the nature of the investigate areas likely to contain fill material and or construction debris, it is expected that utilizing test pits will be most successful for site characterization. To meet the objectives test pits will be placed along two areas between the former Mill buildings and the Kalamazoo River banks in the areas of the elevated PCB concentrations and oil impacted soil. Test pits will be advanced to either native soils or the water table using an excavator taking care to minimize site disturbances to the extent practical. These test pits will be placed to attempt to identify the source and the extent of the oily impacted soil and the elevated PCB concentrations. Additional test pits may be added based upon field observations including odors or other visual signs of contamination. If an additional test pit (or more) are needed or conditions limit the use of test pit excavating equipment, the U.S. EPA project manager, or designated alternate, will be contacted to discuss the situation and to agree on a course of action.

Plainwell Mill RI/FS – Phase I Groundwater Assessment – To meet the objectives groundwater wells will be place around the perimeter of the Plainwell Mill property and specifically targeting well location down gradient of historical facility operations. The wells need to provide important subsurface information such as how the groundwater interacts with the surface water, the groundwater flow direction onsite, additional information regarding subsurface soils, and any shallow groundwater impacts. The results from these wells will assist in targeting areas that may require further investigation. Additional wells may be placed during later phases of the Plainwell Mill RI/FS. If conditions limit the installation of wells or well locations, the U.S. EPA project manager, or designated alternate, will be contacted to discuss the situation and to agree on a course of action.

How much data are needed (number of samples for each analytical group, matrix, and concentration)?

Phase 2 of Addendum No. 1 (Test Pits) – A minimum of 15 test pits will be excavated and sampled for PCBs and TPH (DRO/GRO) in soils. Up to an additional 5 samples will be collected and submitted for RCRA metals, VOCs, and PAHs in soil. Pooled liquids in the test pit may also be sampled, but results would be used for screening purposes only. In addition, if free product is encountered a sample may be collected for determination of physical properties such as specific gravity and viscosity. In the event that in-field conditions limit the use of test pit excavating equipment (e.g., a backhoe), other tools, such as hand augers or shovels, may be used instead.

Plainwell Mill RI/FS – Phase I Groundwater Assessment – A total of 12 groundwater monitoring wells are to be installed. These wells will be sampled for RCRA metals, VOCs, PCBs, and PAHs. At this time, it is anticipated that the wells will be sampled on a quarterly basis.

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

The data will be collected at the locations shown on Figures 6 of Phase 2 of Addendum No. 1 (Test Pits) and Figure 1 of the Plainwell Mill RI/FS – Phase I Groundwater Assessment (Groundwater Monitoring Wells). The data collection activities for Phase 2 of Addendum No. 1 – Test Pits are scheduled to be performed in September – November 2008.

The groundwater wells associated with the Plainwell Mill RI/FS – Phase I Groundwater Assessment are to be installed and sampled between September – November 2008. Additional sampling will likely continue on a quarterly basis. Sampling procedures are detailed in the Standard Operating Procedures (SOPs), which are included in the Multi-Area Field Sampling Plan, Revision 03.

QAPP Worksheet #11-4
Project Quality Objectives/Systematic Planning Process Statements
(Phase 2 of Addendum No. 1 - Test Pits & Plainwell Mill RI/FS – Phase I Groundwater Assessment)

Who will collect and generate the data? RMT will collect the samples.
How will the data be reported? Data from Phase 2 of Addendum No. 1 – Test Pits will be presented in a summary report documenting the test pit observations and analytical results within 60 days of completion of field activities or receipt of analytical data. The data obtained from the Plainwell Mill RI/FS – Phase I Groundwater Assessment will be provided in the 1st monthly progress report after completion of the field activities or receipt of analytical data. The progress report will include a data interpretation summary.
How will the data be archived? Data will be archived in an appropriate database to ensure that information is readily available, searchable, and compatible with a GIS system.

QAPP Worksheet #12-1
Measurement Performance Criteria (PCBs - Surface Water)

Matrix: Aqueous
Analytical Group: PCBs
Concentration Level: All

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	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)
F-1, F-3	W-1	Precision - Overall	Relative percent difference (RPD) < 50%	Field duplicate	S&A
		Accuracy/Bias	% Recovery (%R) laboratory-generated limits[3]	Surrogate	A
		Accuracy/Bias Contamination	< Reporting limit (RL)	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R laboratory-generated limits[*]	Laboratory control sample (LCS)	A
		Accuracy/Bias and Precision	Retention times, see analytical SOP	Retention time windows	A
		Accuracy/Bias	%R, same as LCS[*]	Matrix spike (MS)[3]	A
		Accuracy/Bias	%R, same as LCS[*]	Matrix spike duplicate (MSD) or laboratory control sample duplicate (LCSD)[3]	A
		Precision	RPD <20%	MS/MSD or LCS/LCSD[3]	A

Footnotes:
[1] Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.
[2] Reference number from QAPP Worksheet #23.
[3] MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.
[*] See Attachments 1-1 and 1-2.
RPD = relative percent difference.

QAPP Worksheet #12-2
Measurement Performance Criteria (Wet Chemistry - Surface Water)

Matrix: Water

Analytical Group: Wet chemistry

Concentration Level: All

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	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)
F-3	W-2, W-3, and W-5	Precision - Overall	RPD < 50%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, calibration, preparation)	S&A
		Accuracy/Bias	%R (90-110)	Continuing calibration verification	A
		Accuracy/Bias	%R (75-125)	MS[3]	A
		Accuracy/Bias	%R (75-125)	MSD[3]	A
		Precision	%R < 20%	MS/MSD[3]	A
		Accuracy/Bias	%R laboratory-generated limit[*]	LCS	A

Footnotes:

[1] Reference number from QAPP Worksheet #21. See the project-specific Field sampling Plan for sampling procedures.

[2] Reference number from QAPP Worksheet #23.

[3] MS and MSD not applicable for TSS.

[*] See Attachment 1-1.

QAPP Worksheet #12-3
Measurement Performance Criteria (PCBs - Soil/Sediment)

Matrix: Soil/Sediment

Analytical Group:[(1)] PCBs

Concentration Level: All

SAMPLING PROCEDURE[(2)]	ANALYTICAL METHOD/SOP[(3)]	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)
F-4	W-4	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias	%R, laboratory-generated limits[(*)]	Surrogate	A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, laboratory-generated limits[(*)]	LCS	A
		Accuracy/Bias and Precision	Retention times, see analytical SOP	Retention time windows	A
		Accuracy/Bias	%R, same as LCS[(*)]	MS[(3)]	A
		Accuracy/Bias	%R, same as LCS[(*)]	MSD or LCSD[(3)]	A
		Precision	%RPD <35%	MS/MSD or LCS/LCSD[(3)]	A

Footnotes:

(1) Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.

(2) Reference number from QAPP Worksheet #23.

(3) MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.

(*) See Attachments 1-1 and 1-2.

QAPP Worksheet #12-4
Measurement Performance Criteria (PCBs – Free Product)

Matrix: Free Product
Analytical Group: PCBs
Concentration Level: All

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA[3]	QC SAMPLE AND/OR ACTIVITY USED TO ASSESS MEASUREMENT PERFORMANCE	QC SAMPLE ASSESSES ERROR FOR SAMPLING(S), ANALYTICAL(A), OR BOTH (S&A)
F-8	S-1	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias	%R, laboratory-generated limits(*)	Surrogate	A
		Accuracy/Bias Contamination	< 2 x MDL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, laboratory-generated limits(*)	LCS	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MS[4]	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MSD or LCSD[4]	A
		Precision	RPD <30%	MS/MSD or LCS/LCSD[4]	A

Footnotes:

[1] Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.

[2] Reference number from QAPP Worksheet #23.

[3] Measurement performance criteria listed are current criteria at the time this document was created, but are subject to change as control limits are updated.

[4] MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.

(*) See Attachments 1-1 and 1-2.

RPD = relative percent difference.

QAPP Worksheet #12-5
Measurement Performance Criteria (TAL Metals – Groundwater)

Matrix: Aqueous
Analytical Group: TAL Metals
Concentration Level: All (Except Low-Level Mercury)

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA[3]	QC SAMPLE AND/OR ACTIVITY USED TO ASSESS MEASUREMENT PERFORMANCE	QC SAMPLE ASSESSES ERROR FOR SAMPLING(S), ANALYTICAL(A), OR BOTH (S&A)
F-8	W-14	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, laboratory-generated limits(*)	LCS	A
		Precision	sample > 5 x QL; RPD ≤ 20% sample ≤ 5 x QL; No limit	Laboratory duplicate	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MS[4]	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MSD or LCSD[4]	A
		Precision	RPD <20%	MS/MSD or LCS/LCSD[4]	A
		Accuracy	%R > 80%	Interference check sample	A
		Accuracy	RPD ≤ 10%	Serial dilution	A

Footnotes:
(1) Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.
(2) Reference number from QAPP Worksheet #23.
(3) Measurement performance criteria listed are current criteria at the time this document was created, but are subject to change as control limits are updated.
(4) MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.
(*) See Attachments 1-1.
RPD = relative percent difference.

QAPP Worksheet #12-6
Measurement Performance Criteria (TAL Metals – Soil)

Matrix: Soil
Analytical Group: TAL Metals
Concentration Level: All (Except Low-Level Mercury)

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA[3]	QC SAMPLE AND/OR ACTIVITY USED TO ASSESS MEASUREMENT PERFORMANCE	QC SAMPLE ASSESSES ERROR FOR SAMPLING(S), ANALYTICAL(A), OR BOTH (S&A)
F-5, F-6	W-14	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, laboratory-generated limits[(*)]	LCS	A
		Precision	sample > 5 x QL; RPD ≤ 20% sample ≤ 5 x QL; No limit	Laboratory duplicate	A
		Accuracy/Bias	%R, laboratory-generated limits[(*)]	MS[4]	A
		Accuracy/Bias	%R, laboratory-generated limits[(*)]	MSD or LCSD[4]	A
		Precision	%RPD <40%	MS/MSD or LCS/LCSD[4]	A
		Accuracy	%R > 80%	Interference check sample	A
		Accuracy	RPD ≤ 10%	Serial dilution	A

Footnotes:
(1) Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.
(2) Reference number from QAPP Worksheet #23.
(3) Measurement performance criteria listed are current criteria at the time this document was created, but are subject to change as control limits are updated.
(4) MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.
(*) See Attachments 1-1.
RPD = relative percent difference.

QAPP Worksheet #12-7
Measurement Performance Criteria (Cyanide - Groundwater)

Matrix: Aqueous
Analytical Group: Cyanide
Concentration Level: All, total

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA[3]	QC SAMPLE AND/OR ACTIVITY USED TO ASSESS MEASUREMENT PERFORMANCE	QC SAMPLE ASSESSES ERROR FOR SAMPLING(S), ANALYTICAL(A), OR BOTH (S&A)
F-8	W-25	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, laboratory-generated limits(*)	LCS	A
		Precision	%RPD <20%	Laboratory duplicate	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MS[4]	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MSD or LCSD[4]	A
		Precision	%RPD <20%	MS/MSD or LCS/LCSD[4]	A

Footnotes:

[1] Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.

[2] Reference number from QAPP Worksheet #23.

[3] Measurement performance criteria listed are current criteria at the time this document was created, but are subject to change as control limits are updated.

[4] MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.

(*) See Attachments 1-1.

RPD = relative percent difference.

QAPP Worksheet #12-8
Measurement Performance Criteria (Cyanide - Soil)

Matrix: Soil
Analytical Group: Cyanide
Concentration Level: All, total

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA[3]	QC SAMPLE AND/OR ACTIVITY USED TO ASSESS MEASUREMENT PERFORMANCE	QC SAMPLE ASSESSES ERROR FOR SAMPLING(S), ANALYTICAL(A), OR BOTH (S&A)
F-8	W-25	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, laboratory-generated limits(*)	LCS	A
		Precision	%RPD <20%	Laboratory duplicate	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MS[4]	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MSD or LCSD[4]	A
		Precision	%RPD <20%	MS/MSD or LCS/LCSD[4]	A

Footnotes:

[1] Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.

[2] Reference number from QAPP Worksheet #23.

[3] Measurement performance criteria listed are current criteria at the time this document was created, but are subject to change as control limits are updated.

[4] MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.

(*) See Attachments 1-1.

RPD = relative percent difference.

QAPP Worksheet #12-9
Measurement Performance Criteria (Low-Level Mercury - Groundwater)

Matrix: Aqueous
Analytical Group: Mercury
Concentration Level: Low-Level

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA[3]	QC SAMPLE AND/OR ACTIVITY USED TO ASSESS MEASUREMENT PERFORMANCE	QC SAMPLE ASSESSES ERROR FOR SAMPLING(S), ANALYTICAL(A), OR BOTH (S&A)
F-8	C-4	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, laboratory-generated limits(*)	LCS	A
		Precision	RPD	Laboratory duplicate	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MS[4]	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MSD or LCSD[4]	A
		Precision	%RPD <24%	MS/MSD or LCS/LCSD[4]	A

Footnotes:

(1) Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.

(2) Reference number from QAPP Worksheet #23.

(3) Measurement performance criteria listed are current criteria at the time this document was created, but are subject to change as control limits are updated.

(4) MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.

(*) See Attachments 1-1.

RPD = relative percent difference.

QAPP Worksheet #12-10
Measurement Performance Criteria (SVOCs - Groundwater)

Matrix: Aqueous
Analytical Group: SVOCs
Concentration Level: All

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA[3]	QC SAMPLE AND/OR ACTIVITY USED TO ASSESS MEASUREMENT PERFORMANCE	QC SAMPLE ASSESSES ERROR FOR SAMPLING(S), ANALYTICAL(A), OR BOTH (S&A)
F-8	W-15	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, Laboratory-generated limits(*)	LCS	A
		Accuracy/Bias	%R, Laboratory-generated limits(*)	MS[4]	A
		Accuracy/Bias	%R, Laboratory-generated limits(*)	MSD or LCSD[4]	A
		Precision	%RPD <20%	MS/MSD or LCS/LCSD[4]	A
		Accuracy/Bias	%R, Laboratory-generated limits(*)	Surrogates	A
		Accuracy/Bias	+100%, -50% of ICAL	Internal standards	A
		Accuracy/Bias	Established mass limits	DFTPP Tuning	A

Footnotes:

(1) Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.

(2) Reference number from QAPP Worksheet #23.

(3) Measurement performance criteria listed are current criteria at the time this document was created, but are subject to change as control limits are updated.

(4) MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.

(*) See Attachments 1-1 and 1-2.

RPD = relative percent difference.

QAPP Worksheet #12-11
Measurement Performance Criteria (SVOCs - Soil)

Matrix: Soil
Analytical Group: SVOCs
Concentration Level: All

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA[3]	QC SAMPLE AND/OR ACTIVITY USED TO ASSESS MEASUREMENT PERFORMANCE	QC SAMPLE ASSESSES ERROR FOR SAMPLING(S), ANALYTICAL(A), OR BOTH (S&A)
F-5, F-6	W-15	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, Laboratory-generated limits(*)	LCS	A
		Accuracy/Bias	%R, Laboratory-generated limits(*)	MS[4]	A
		Accuracy/Bias	%R, Laboratory-generated limits(*)	MSD or LCSD[4]	A
		Precision	%RPD <40%	MS/MSD or LCS/LCSD[4]	A
		Accuracy/Bias	%R, Laboratory-generated limits(*)	Surrogates	A
		Accuracy/Bias	+100%, -50% of ICAL	Internal standards	A
		Accuracy/Bias	Established mass limits	DFTPP Tuning	A

Footnotes:

(1) Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.

(2) Reference number from QAPP Worksheet #23.

(3) Measurement performance criteria listed are current criteria at the time this document was created, but are subject to change as control limits are updated.

(4) MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.

(*) See Attachments 1-1 and 1-2.

RPD = relative percent difference.

QAPP Worksheet #12-12
Measurement Performance Criteria (VOCs - Groundwater)

Matrix: Aqueous
Analytical Group: VOCs
Concentration Level: All

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA[3]	QC SAMPLE AND/OR ACTIVITY USED TO ASSESS MEASUREMENT PERFORMANCE	QC SAMPLE ASSESSES ERROR FOR SAMPLING(S), ANALYTICAL(A), OR BOTH (S&A)
F-8	W-21	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, laboratory-generated limits(*)	LCS	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MS[4]	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MSD or LCSD[4]	A
		Precision	%RPD <20%	MS/MSD or LCS/LCSD[4]	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	Surrogates	A
		Accuracy/Bias	+100%, -50% of ICAL	Internal standards	A
		Accuracy/Bias	Established mass limits	BFB Tuning	A

Footnotes:

(1) Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.

(2) Reference number from QAPP Worksheet #23.

(3) Measurement performance criteria listed are current criteria at the time this document was created, but are subject to change as control limits are updated.

(4) MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.

(*) See Attachments 1-1 and 1-2.

RPD = relative percent difference.

QAPP Worksheet #12-13
Measurement Performance Criteria (VOCs - Soil)

Matrix: Soil
Analytical Group: VOCs
Concentration Level: All

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA[3]	QC SAMPLE AND/OR ACTIVITY USED TO ASSESS MEASUREMENT PERFORMANCE	QC SAMPLE ASSESSES ERROR FOR SAMPLING(S), ANALYTICAL(A), OR BOTH (S&A)
F-5, F-6	C-5	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, laboratory-generated limits(*)	LCS	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MS[4]	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MSD or LCSD[4]	A
		Precision	%RPD <40%	MS/MSD or LCS/LCSD[4]	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	Surrogates	A
		Accuracy/Bias	Established mass limits	BFB Tuning	A

Footnotes:

(1) Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.

(2) Reference number from QAPP Worksheet #23.

(3) Measurement performance criteria listed are current criteria at the time this document was created, but are subject to change as control limits are updated.

(4) MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.

(*) See Attachments 1-1 and 1-2.

RPD = relative percent difference.

QAPP Worksheet #12-14
Measurement Performance Criteria (DRO & GRO - Soil)

Matrix: Soil
Analytical Group: DRO, GRO
Concentration Level: All

SAMPLING PROCEDURE[1]	ANALYTICAL METHOD/SOP[2]	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA[3]	QC SAMPLE AND/OR ACTIVITY USED TO ASSESS MEASUREMENT PERFORMANCE	QC SAMPLE ASSESSES ERROR FOR SAMPLING(S), ANALYTICAL(A), OR BOTH (S&A)
F-5, F-6	W-22 (DRO) W-23 (GRO)	Precision-Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R, laboratory-generated limits(*)	LCS	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MS[4]	A
		Accuracy/Bias	%R, laboratory-generated limits(*)	MSD or LCSD[4]	A
		Precision	%RPD <10%	MS/MSD or LCS/LCSD[4]	A
		Accuracy/Bias	%R, Laboratory-generated limits (*)	Surrogate	A

Footnotes:

(1) Reference number from QAPP Worksheet #21. See the project-specific Field Sampling Plan for sampling procedures.

(2) Reference number from QAPP Worksheet #23.

(3) Measurement performance criteria listed are current criteria at the time this document was created, but are subject to change as control limits are updated.

(4) MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD is supplied.

(*) See Attachments 1-1 and 1-2.

RPD = relative percent difference.

QAPP Worksheet #13-1
Secondary Data Criteria and Limitations (12th Street Landfill)

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 residuals sampling data	June and Sept. 1987 and January, 1989. MDNR and Mill Staff, 24 soil and residual samples outside berm. (Discussed in G&M, 1994a).	Plainwell Paper Co.; KRSG	Remedial design	U.S. EPA approval
Electromagnetic conductivity and magnetometer survey data	G&M, 1991. "12th Street Landfill Geophysical Investigation, Plainwell, Michigan." Letter Report by Geraghty and Miller, Inc. October 11, 1991.	Plainwell Paper Co.; KRSG	Remedial design	U.S. EPA approval
Soil and residuals sampling data	G&M, 1994a. "Test Pit Investigation, Technical Memorandum, 12th Street Landfill Operable Unit, Plainwell, Michigan, Allied Paper Inc./Portage Creek/Kalamazoo River Superfund Site." Geraghty and Miller, Inc. February 18, 1994.	Plainwell Paper Co.; KRSG	Remedial design	U.S. EPA approval
Sediment probing data; soil and residuals sampling data, groundwater sampling data	G&M, 1994b. Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund site Remedial Investigation/Feasibility Study, Technical Memorandum 8, 12th Street Landfill Operable Unit, Plainwell, Michigan. May 31, 1994.	Plainwell Paper Co.; KRSG	Site characterization	U.S. EPA approval(1)
Groundwater sampling data	G&M, 1996a. "Remedial Investigation Addendum I, 12th Street Landfill Operable Unit, Plainwell, Michigan, Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site." March 26, 1996.	Plainwell Paper Co.; KRSG	Remedial design	U.S. EPA approval
Summary of existing data; residuals sampling data	G&M, 1996b. "Remedial Investigation Report, 12th Street Landfill Operable Unit, Plainwell Michigan, Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site." Geraghty and Miller, Inc. December 20, 1996.	Plainwell Paper Co.; KRSG	Remedial design	U.S. EPA approval

QAPP Worksheet #13-1
Secondary Data Criteria and Limitations (12th Street Landfill)

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
Geotechnical data	BBL, 2001a. "Geotechnical Sample Analytical Data, 12th Street Landfill Operable Unit." Transmittal Letter with Attachments. Blasland, Bouck & Lee, Inc. June 25, 2001.	KRSG	Remedial design	U.S. EPA approval
Sediment probing data	BBL, 2001b, as reported in final data summary report soil/sediment sampling results pre-design sampling. 12th Street Landfill, Operable Unit #4, Allied Paper, Inc./Portage Creek/Kalamazoo River Site, Plainwell, Michigan. July 2004.	KRSG	Site characterization	U.S. EPA approval(1)
Wetland delineation	CDM, 2002. "Michigan Department of Environmental Quality, Kalamazoo River and Portage Creek Wetland Delineation Study," Camp Dresser and McKee. January 2002.	MDEQ	Remedial design	U.S. EPA approval
Sediment probing data	U.S. EPA, 2004. Final data summary report soil/sediment sampling results pre-design sampling. 12th Street Landfill, Operable Unit #4, Allied Paper, Inc./Portage Creek/Kalamazoo River Site, Plainwell, Michigan. July 2004.	U.S. EPA	Site characterization	U.S. EPA approval(1)

Footnote:

(1) Data and observations collected during the sediment excavation will take precedence.

QAPP Worksheet #13 -2
Secondary Data Criteria and Limitations
(Plainwell Mill)

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
Sediment coring observations and survey results	RMT, July 2007. Visual Assessment of the Plainwell Mill Banks.	RMT July 2007	Site Characterization	U.S. EPA approval(1)
Prior investigations	BBL, 1996. Technical Memorandum 15: Mill Investigations	BBL 1996	Site Characterization	Data are historical and site conditions may have changed.
	ERM, 1997. Simpson Plainwell Paper Company Phase I environmental site assessment and Phase II investigation, Plainwell, Michigan	ERM 1997		
	Plainwell Paper, 1999 & 2000. Letter to Michigan Department of Environmental Quality.	Simpson Plainwell Oil spill clean-up samples 1999 & 2000		
	CDM, 2001. Plainwell Paper Gray Seam Investigation.	CDM October 2001		
	Weston, 2002. Removal Assessment Report for Allied Paper – Kalamazoo River Site, Otsego/Plainwell.	Weston July and August 2001		
	BBL, 2003. PCB Data for the Plainwell Impoundment River Bank Soil Samples.	BBL October 2003		
	FTCH, 2006. Phase II Environmental Site Assessment, Former Plainwell Paper Mill.	FTCH October 2006		
	RMT, 2008. Plainwell Mill Banks Emergency Action Documentation Report.	RMT Data available, but report has not yet been submitted.		

Footnote:
(1) Data and observations collected during the sediment excavation will take precedence.

QAPP Worksheet #14
Summary of Project Tasks

SAMPLING AND ANALYSIS TASKS	
Emergency Response Activities in the Former Powerhouse Discharge Channel at the 12th Street Landfill (Operable Unit No. 4)	
Sampling tasks:	Sampling tasks will be performed as follows: ▪ Pre-excavation bathymetry measurements and sediment sampling to plan extent of sediment excavation (*i.e.*, analytical data for polychlorinated biphenyls (PCBs), visual characterization, and engineering data) ▪ Resuspension monitoring and control, including surface water sampling and field turbidity measurements ▪ Dewatering system discharge monitoring ▪ Post-excavation confirmation sampling for PCBs in channel sediment ▪ Global positioning system (GPS)–based survey of excavation limits and sampling locations ▪ Digital photographs
Analysis tasks:	Analytical samples will be processed, prepared, and analyzed, as follows: ▪ Resuspension monitoring and control (surface water) — PCBs by WATS ▪ Dewatering system discharge monitoring (surface water) — PCBs, total suspended solids (TSS), and phosphorus by WATS ▪ Post-excavation confirmation sampling (sediment) — PCBs by WATS
Quality control tasks:	The samples will be collected and processed, and the laboratory waste will be disposed of as described in the laboratory SOPs (Attachment 1-5). QA samples will be collected as described in Worksheet #26.
Secondary data:	See Worksheets #13-1 and #13-2.

QAPP Worksheet #14 *(continued)*
Summary of Project Tasks

SAMPLING AND ANALYSIS TASKS	
Predesign Investigations for the Remedial Design for the 12th Street Landfill (Operable Unit No. 4)	
Sampling tasks:	Field data collection activities during the predesign investigations will include: ▪ Visual information obtained by the advancement of test pits ▪ Visual information obtained by the advancement of Geoprobe® borings ▪ Gas concentrations (methane, carbon dioxide, and oxygen) measured in existing monitoring wells and in the Geoprobe® locations. Pressure measured in existing monitoring wells.
Analysis tasks:	No analytical data will be collected.
Quality control tasks:	No analytical data will be collected.
Secondary data:	See Worksheet #13-1.
Phase 2 of Addendum No. 1 – Test Pits & Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment	
Sampling tasks:	Sampling tasks will be performed as follows: **Phase 2 of Addendum No. 1** Approximately 15 test pits will be excavated along the banks of the Plainwell Mill buildings. ▪ Test pit excavation to determine extent of oil impacted soil and elevated concentrations of PCBs (*i.e.*, analytical data for polychlorinated biphenyls (PCBs) & total petroleum hydrocarbons, visual characterization, and engineering data) ▪ Additional soil samples will be collected from the test pits for RCRA metals, VOCs, and PAHs ▪ If free product is observed a sample will be collected for general characteristics ▪ Global positioning system (GPS)–based survey of excavation limits and sampling locations ▪ Digital photographs **Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment** Approximately 12 groundwater wells and three staff gages will be installed. ▪ Collect groundwater samples for RCRA metals, VOCs, PAHs, and PCBs ▪ Collect water elevations from three surface water staff gages (Two in the Kalamazoo River and One in the Mill Race) ▪ Global positioning system (GPS)–based survey of excavation limits and sampling locations ▪ Digital photographs

QAPP Worksheet #14 *(continued)*
Summary of Project Tasks

Analysis tasks:	Analytical samples will be processed, prepared, and analyzed, as follows: **Phase 2 of Addendum No. 1 - Test Pits Soil Samples** ▪ PCBs by WATS ▪ RCRA Metals by WATS ▪ PAHs by WATS ▪ VOCs by Columbia Analytical Services ▪ Free Product Characterization (Specific Gravity & Viscosity) – Summit Technologies, Inc. ▪ Total petroleum hydrocarbons (DRO/GRO) by WATS **Plainwell Mill RI/FS – Initial Groundwater Assessment (Groundwater Samples)** ▪ PCBs by WATS ▪ RCRA Metals by WATS o Hg by Columbia Analytical Services ▪ PAHs by WATS ▪ VOCs by Columbia Analytical Services
Quality control tasks:	The samples will be collected and processed, and the laboratory waste will be disposed of as described in the laboratory SOPs (Attachment 1-5). QA samples will be collected as described in Worksheet #26.
Secondary data:	See Worksheets #13-1 and #13-2.

QAPP Worksheet #14 *(continued)*
Summary of Project Tasks

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Data management tasks:	▪ **Field data reduction** - Raw data from field measurements and sample collection activities will be recorded as specified in the FSP. If the data are to be used in the project reports, they will be reduced or summarized, and the method of reduction will be documented in the report. With the exception of the temperature correction for specific conductance, no calculation will be involved in field data reduction. Only direct-reading instrumentation will be employed in the field. The On-Site Coordinator or designee will proofread all forms and notebooks to determine if transcription errors have been made by the field crew. ▪ **Laboratory data reduction** - WATS will perform in-house analytical data reduction under the direction of the laboratory QA/QC Manager. The laboratory QA/QC Manager will be responsible for assessing data quality and advising of any data that were rated "preliminary" or "unacceptable" or of other notations that would caution the data user of possible unreliability. Data reduction, by the laboratory, will be conducted as follows: — The analysts who produced the laboratory data will first conduct a systematic review (Level 1 Review). — An experienced peer, supervisor, or designee will examine the data (the Level 2 Review) to ensure that the Level 1 review has been completed correctly and thoroughly. Following the Level 2 review, the data will be turned over to the Laboratory Project Manager for a third-level review. — The Project Manager will review the data for completeness and attainment of quality control criteria as outlined in the U.S. EPA methods and for overall reasonableness. — The Project Manager will verify the accuracy and completeness of the final reports. — The Laboratory QA/QC Manager and the supervisor of the pertinent analytical section, in conjunction with the RMT Laboratory Coordinator, will decide whether any sample reanalysis is required. — Data reduction procedures are included in the U.S. EPA-approved methods and associated laboratory SOPs. ▪ **Field data reporting** - Field data reporting will consist of field logs and calibration and measurement records documenting site activities as described in the FSP and on the sample Chain-of-Custody Records.

QAPP Worksheet #14 *(continued)*
Summary of Project Tasks

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Data management tasks: (***continued***)	▪ **Laboratory data reporting** - The analytical laboratories will prepare and retain full analytical and QC documentation. Such retained documentation need not be on hard (paper) copy, but may be in other storage media (*e.g.*, computer diskette or magnetic tape). As needed, WATS will supply a hard copy of the retained information. WATS will provide the following information in each analytical data package submitted: — Dated cover sheets, signed by the WATS project manager, listing a laboratory batch number; the analyses performed; the number of samples and respective matrices; the project name and number; narrative comments describing deviations from intended analytical strategy, and any problems encountered in analysis; a discussion of any laboratory quality control checks that failed to meet project criteria; and the signature of the laboratory QA/QC Manager — Tabulated results of inorganic and organic compounds identified and quantified, including sample preparation and analysis dates, and cross-references of laboratory and field sample identification numbers — Analytical results for QC sample spikes and sample duplicates; initial and continuing calibration verifications of standards and blanks; standard procedural blanks; laboratory control samples; and the data produced by ICP interference check samples, as appropriate for the specified analyses — Tabulation of Method Detection Limits, as appropriate — Raw data system printouts (or legible photocopies) identifying the date of analyses, the mass spectra tuning data, the name of the analyst, the parameters determined, the initial and continuing calibration, the calibration verification summary, the method blanks, the sample and any dilutions, sample duplicates and spikes, chromatograms, GC/MS spectra, computer printouts, internal standard area and RT summary, cleanup information, control samples, ICP outputs, and inter-element correction data WATS will provide the following information for the indicator parameters in each analytical data package submitted: — Dated cover sheets listing a laboratory batch number; the analyses performed; the number of samples and respective matrices; project name and number; narrative comments describing deviations from intended analytical strategy, and problems encountered in analysis; discussion of any laboratory quality control checks that failed to meet project criteria; and the signature of the Laboratory QA Manager — Copies of bench sheets that include summaries of initial calibration and continuing calibration check results — Summary of QC sample analysis (spikes, duplicates, laboratory control samples, and blanks). A report will be prepared containing a QA/QC section summarizing the quality of the data. The QA report prepared by the RMT will address the assessment of data precision, accuracy, completeness, and comparability; the results of performance audits, if any; the results of system audits; any reported nonconformances; any significant QA/QC problems and recommended solutions; the results of corrective actions since the last report; and approved revisions to the QA/QC processes. The report will indicate whether the QA objectives were met and whether the data can be used for the intended purposes based on an evaluation of compliance with control limits, the results of audits, and compliance with the procedures specified in the QAPP and the FSP.

QAPP Worksheet #14 *(continued)*
Summary of Project Tasks

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Documentation and records:	Appropriate records will be maintained to provide adequate documentation of the entire data generation process, including field sampling and laboratory analysis. ▪ **Field documentation -** Field personnel will develop and retain comprehensive records of field activities, including field sampling, field analysis, and sample Chain-of-Custody, to allow a reconstruction of field events and sample handing during data review and interpretation. ▪ **Laboratory project files -** WATS will maintain a file for pertinent project information, including Chain-of-Custody Records; other custody documents (air bills, etc.); work orders; Sample Receipt Acknowledgment Forms, if any; instrument detection limit and control limit tabulations; all raw analytical data on bench sheets; laboratory data; and project communication records. Such retained documentation need not be on hard (paper) copy, but may be in other storage media (*e.g.*, computer diskette or magnetic tape). As needed, WATS will supply a hard copy of the retained information. ▪ **Laboratory notebooks -** Logbooks, bench sheets, instrument notebooks, and instrument printouts will be retained as part of the permanent laboratory record, including the associated quality controls. Each page in the laboratory logbooks and bench sheets will be signed and dated by the analyst, and errors will be crossed out in indelible ink. System printouts of raw inorganic and organic data will include dates of analyses; analyst's name; parameters determined; calibration curve; calibration verifications; method blanks; sample number and dilutions performed; and sample duplicates, spikes, and control samples. Internal laboratory QC sample results will be indicated on the analytical bench sheets and will include sample spikes, sample duplicates, initial and continuous calibration verification of standards and blanks, standard procedural blanks, laboratory control samples, ICP serial dilutions, and ICP interference check samples. ▪ **Computer and hard copy storage -** All electronic files and deliverables will be retained by the laboratory for no less than 5 years. Weyerhaeuser, or its designated representatives, will retain copies of the analytical data reports according to the requirements of the laboratory QA Manual. All field records will be kept in the central project file at the RMT office at 744 Heartland Trail, Madison, Wisconsin; and records will be included in project reports, as appropriate, or upon request by the U.S. EPA RPM. ▪ **Field data reporting -** Field data reporting will consist of field notebooks and logs, photographs, boring logs, calibration and measurement records, and Chain–of-Custody (COC) documentation, including field quality control samples that will be collected to assess the quality of the analytical data and to evaluate sampling and analytical reproducibility. Field records will be reviewed by the Project Coordinator for consistency with the planned activities, and any concerns will be discussed with the Field Sampling Coordinator. Field performance and field system audits will also be performed, as discussed below and in Worksheets #31 and 32. ▪ **Laboratory data reporting -** Analytical data for this project will be reported in both an electronic data deliverable (EDD) and an analytical data package. The EDD will be generated by WATS and will be used by RMT to facilitate loading the analytical data into the project database. The Laboratory QA/QC Manager will perform a final review of the report summaries and case narratives to determine if the report meets project requirements. The task of reporting laboratory data to the U.S. EPA will begin after the data review activity has been concluded. The validated analytical data will be provided to the U.S. EPA in accordance with the project schedule (Worksheet #16). In addition to the COC Record, WATS will prepare and provide a full "CLP-like" data package, including the following:

QAPP Worksheet #14 *(continued)*
Summary of Project Tasks

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Documentation and records: (***continued***)	— **Case narrative -** date of issuance; laboratory analysis performed; any deviations from required analytical methods; laboratory sample lot numbers; numbers of samples and respective matrices; QC procedures used and references to the acceptance criteria; laboratory report table of contents; project name and number; condition of samples upon receipt; dates of extraction, preparation, and analysis; discussion of whether or not sample holding times were met; discussion of technical problems or other observations that may have created analytical difficulties; discussion of any laboratory QC checks that failed to meet project criteria; signature of the laboratory Project Manager, and copies of the COC Records — **Chemistry data package -** run log, summary page indicating dates of analyses for samples and laboratory QC checks, cross-referencing of laboratory sample to project sample identification numbers**,** adequately described data qualifiers, sample preparation and analysis methods, sample results, matrix spike and matrix spike duplicate (MS/MSD) recoveries, laboratory control sample recoveries, method blank results, and surrogate recoveries Soil sample analytical data generated by the laboratory will be reported in micrograms per kilogram (µg/kg) on a dry-weight basis. Groundwater data will be reported in micrograms per liter (µg/L). Results between the laboratory Method Detection Limit (MDL) and the Quantitation Limit (QL) will be reported. Data retained in the project database may be converted to units other than those reported by the laboratories. Sample results will not be corrected for contamination found in laboratory blanks. However, sample results may be qualified as not detected based on laboratory, field, and/or trip blank contamination. WATS will provide the following information in each analytical data package submitted: ▪ Dated cover sheets, signed by the WATS Project Manager, listing a laboratory batch number; the analyses performed; the number of samples and the respective matrices; the project name and number; narrative comments describing deviations from intended analytical strategy, and problems encountered in analysis; a discussion of any laboratory quality control checks that failed to meet project criteria; and the signature of the laboratory QA/QC Manager ▪ Tabulated results of inorganic and organic compounds identified and quantified, including sample preparation and analysis dates, and cross-references of laboratory and field sample identification numbers ▪ Analytical results for QC sample spikes, sample duplicates, and blanks; initial and continuing calibration verifications of standards and blanks; standard procedural blanks; laboratory control samples; and the data produced by inductively coupled plasma (ICP) interference check samples, as appropriate for the specified analyses ▪ Tabulation of Method Detection Limits, as appropriate ▪ Raw data system printouts (or legible photocopies) identifying dates of analyses, mass spectra tuning data, name of analyst, parameters determined, initial and continuing calibration, calibration verification summary, method blanks, sample and any dilutions, sample duplicates and spikes, chromatograms, gas chromatograph/mass spectrophotometer (GC/MS) spectra, computer printouts, internal standard area and retention time (RT) summary, cleanup information, control sample results, ICP outputs, and inter-element correction data

QAPP Worksheet #14 *(continued)*
Summary of Project Tasks

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Assessment/Audit tasks:	Performance and system audits will be completed in the field and laboratory, as described below and in Worksheets #31 and 32. ▪ **Field audits** - The Project Coordinator will monitor day-to-day field performance through daily communications with the on-site Field Sampling Coordinator and the Construction Manager. In addition, field performance audits and field system audits will be performed, as follows: — **Field performance audits** - Field performance audits will be conducted in order to confirm that the activities are being performed according to the established plans. The field performance audit(s) will be performed by the Senior Consultant QA Manager (or her designee), at a frequency that is appropriate for the field activities being performed. The audit(s) will include a discussion of the project progress with the Project Coordinator and/or the review of field reports, as appropriate. The Senior Consultant QA Manager will record and document any observations made during field system audits, and will discuss the audit and any recommended changes/deviations to the field procedures with the Project Coordinator. — **Field system audits -** Field system audits will be performed by the Data QA Manager, including a review of rinse and trip blank data to identify potential deficiencies in field sampling and decontamination procedures, and a comparison of the scheduled QA/QC activities described in this QAPP with the QA/QC procedures being performed on the project. Field system audits will be performed at a frequency appropriate for the field activities. The Data QA Manager will record and document any observations made during field system audits, and will discuss the audit and any recommended changes/deviations to the field procedures with the Project Coordinator. ▪ **Laboratory audits -** Laboratory audits will be performed, as follows: — **Internal audits** - The Laboratory QA/QC Manager (or his designee) will conduct internal laboratory audits periodically. This will include an overall evaluation of the performance of laboratory staff and a comparison of laboratory procedures with the laboratory QA Manual and SOPs. Results of the audits will be summarized and distributed to appropriate laboratory staff. — **External audits -** The Data QA Manager will review the laboratory QA Manual and applicable SOPs, and will discuss laboratory procedures with the Laboratory QA/QC Manager prior to the start of project sampling. The Data QA Manager will record and document any observations made during the review. In addition, as a participant in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification. Audits include a review of sample handling and tracking documentation, analytical methodologies, analytical supportive documentation, and final reports. The audit findings are documented and submitted to the laboratory for corrective action, if necessary. ▪ **Corrective action** - Corrective actions are required when field or analytical data are not within the objectives specified in this QAPP, as follows:

QAPP Worksheet #14 *(continued)*
Summary of Project Tasks

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Assessment/Audit tasks: (*continued*)	— **Field measurement corrective action** - Corrective action in the field may be necessary when the sample network is changed (*i.e.*, more/fewer samples, sampling locations other than those specified in the FSP, etc.), or when sampling procedures and/or field analytical procedures require modification in response to unexpected conditions. Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances or deficiencies of any activity or issued document by reporting the situation to the RMT on-site Field Sampling Coordinator or designee. The Field Sampling Coordinator will assess the suspected problems in consultation with the Project Coordinator or Data QA Manager or designee, and will assist in making a decision based on the potential for the situation to impact the data quality. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, the On-Site Coordinator will initiate a nonconformance report. If appropriate, the RMT Field Sampling Coordinator will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed. — **Laboratory corrective action** - Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. Corrective actions may be necessary if any of the following occur: ▪ QC data are outside the warning or acceptable windows for precision and accuracy. ▪ Blanks contain target analytes above acceptable levels. ▪ Undesirable trends are detected in spike recoveries or the RPD between duplicates. ▪ There are unusual changes in detection limits. ▪ Deficiencies are detected by the QA Manager during internal or external audits or from the results of performance evaluation samples. ▪ Inquiries concerning data quality are received. Corrective actions should be timely, and they should determine the root cause and evaluate any propagation of the error or problem. The investigative action taken is somewhat dependent on the analysis and the event. Corrective action in the laboratory may occur prior to, during, or after the initial analysis. Corrective action is under the supervision of the Laboratory QA/QC Manager. Following a consultation with laboratory scientists, technicians, and team leaders, it may be necessary for the QA Manager to approve the implementation of the corrective action. Some conditions during or after analysis may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, automatic reinjection/reanalysis when certain quality control criteria are not met, etc. WATS' corrective action procedures are documented in Laboratory SOPs specifying corrective action to be taken when an analytical error is discovered or the analytical system is found to be out of control.

QAPP Worksheet #14 *(continued)*
Summary of Project Tasks

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Assessment/Audit tasks: (***continued***)	Depending on the problem, the corrective action employed may be formal or informal. On-the-spot actions are used to correct minor problems, such as recalibration, retuning, or a minor repair (*e.g.*, replacement of a minor part) of a malfunctioning instrument or the correction of poor analytical technique being used. Corrective action procedures may be handled at the bench level by the analyst, who reviews the preparation or extraction procedure that was used for possible errors, and checks the instrument calibration, spike, and calibration mixes, and the instrument sensitivity. These occurrences are documented in the appropriate injection, run, or analysis logbooks. Similarly, routine instrument maintenance, malfunctions, and power failures are also documented in the appropriate instrument maintenance logbooks. If the problem persists or cannot be identified, the matter may be referred to the laboratory team leader, and/or QA/QC Manager for further investigation. Occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated will be properly documented. The corrective action procedure will be discussed with the Laboratory Project Manager, and full documentation of the corrective action procedure, whether resolved or not, will be placed in the laboratory project file. Corrective actions specific to analytical methods are discussed in the operational-specific SOPs. The U.S. EPA RPM or the RMT Data QA Manager may request corrective action for any nonconformance identified by audits or data validation. — **Corrective action during data validation and data assessment** - The need for corrective actions may be identified during data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory. Data validation corrective actions may include notification of the laboratory of incomplete or erroneous reports and a request for issuance of corrected versions. When the Data QA Manager identifies a corrective action situation, the Project Coordinator will approve the implementation of corrective action, including possible resampling. The RMT Data QA Manager will notify the laboratory of incomplete or erroneous reports and will request the issuance of corrected versions. All corrective actions will be documented. Final summary data tables will not be issued until all data have been validated and all corrections have been made. Corrective action may include the following: ▪ Reanalysis of samples, if holding time requirements permit ▪ Resampling and analysis ▪ Evaluation and amendment of sampling procedures ▪ Evaluation and amendment of analytical procedures ▪ Acceptance of data and acknowledgment of the level of uncertainty
Data Review tasks:	See Worksheets #36 and 37.

QAPP Worksheet #15-1
Reference Limits and Evaluation (Surface Water)

ANALYTE	CAS NUMBER	PROJECT ACTION LIMIT[1]	WATER (µg/L)[2]	
			LABORATORY MDL	LABORATORY RL
PCBs (U.S. EPA 608)[3]				
Aroclor - 1016	12674-11-2	--	0.10	0.2
Aroclor - 1221	11104-28-2	--	--	0.2
Aroclor - 1232	11141-16-5	--	--	0.2
Aroclor - 1242	53469-21-9	--	--	0.2
Aroclor - 1248	12672-29-6	--	--	0.2
Aroclor - 1254	11097-69-1	--	--	0.2
Aroclor - 1260	11096-82-5	--	0.10	0.2
Total PCBs	1336-36-3	0.2 µg/L	--	0.2
Inorganics (Wet Chemistry)[4]				
TSS (U.S. EPA 160.2)	--	30-45 mg/L	--	5.0 mg/L[5]
Total phosphorus as P (U.S. EPA 365.2)	--	--	--	0.01 mg/L

Footnotes:
(1) Standards are consistent with the Multi-Area Quality Assurance Project Plan (Rev. 00) For the Kalamazoo River Study Group (Arcadis BBL, 2007b), which are from the MDEQ's Substantive Requirements Document for the Plainwell Dam Superfund Site, dated February 13, 2007. The TSS standards are on a monthly and daily basis, respectively.
(2) Concentrations are in µg/L, except where noted.
(3) U.S. EPA. *Appendix A to Part 136 Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater Method 608—Organochlorine Pesticides and PCBs*. July 1995.
(4) U.S. EPA. *Methods for Chemical Analysis of Water and Wastes.* EPA/600/4-79/020. EMSL-Cincinnati. 1983.
(5) The actual Reporting Limit will depend on the volume of sample filtered.

Notes:
RL = reporting limit.
MDL = Method Detection Limit.
µg/L = micrograms per liter.
mg/L = milligrams per liter.
PCBs = polychlorinated biphenyls.

QAPP Worksheet #15-2
Reference Limits and Evaluation (Soil/Sediment)

PARAMETER[1][4]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µg/kg)			PART 201 TDLs (µg/kg)	QUANTITATION LIMIT[2] (µg/kg)	METHOD DETECTION LIMIT[2] (µg/kg)
METALS AND CYANIDE							
Aluminum	7429905	1,000	-	1,000,000,000	1,000	20,000	30
Antimony	7440360	4,300	-	49,000,000	300	10,000	7
Arsenic	7440382	4,600	-	2,000,000	100	3,000	3
Barium	7440393	760,567	-	1,000,000,000	1,000	500	2
Beryllium	7440417	51,000	-	1,000,000,000	500	500	2
Cadmium	7440439	5,280	-	230,000,000	200	1,000	1
Chromium (III)	16065831	150,000,000	-	4,520,736,000	2,000	1,000	3
Chromium (VI)	18540299	3,300	-	140,000,000	2,000	1,000	3
Cobalt	7440484	800	-	48,000,000	500	1,000	1
Copper	7440508	112,839	-	1,000,000,000	1,000	2,000	3
Iron	7439896	6,000	-	1,000,000,000	5,000	5,000	70
Lead	7439921	400,000	-	100,000,000	1,000	3,000	1
Magnesium	7439954	8,000,000	-	6,700,000,000	4,000	10,000	20
Manganese	7439965	1,000	-	180,000,000	1,000	500	2
Mercury (total)	7439976	50	-	20,000,000	50	2,000	NA[5]
Nickel	7440020	100,000	-	1,000,000,000	1,000	5,000	6
Selenium	7782492	400	-	130,000,000	200	3,000	10
Silver	7440224	100	-	200,000,000	100	1,000	4
Sodium	17341252	2,500,000	-	1,000,000,000	10,000	50,000	100
Thallium	7440280	2,300	-	15,000,000	500	3,000	1
Vanadium	7440622	72,000	-	1,000,000,000	1,000	1,000	20
Zinc	7440666	254,726	-	1,000,000,000	1,000	1,000	30
Cyanide	57125	100	-	250,000	100	400	NA[5]

QAPP Worksheet #15-2
Reference Limits and Evaluation (Soil/Sediment)

PARAMETER[1][4]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µg/kg)			PART 201 TDLs (µg/kg)	QUANTITATION LIMIT[2] (µg/kg)	METHOD DETECTION LIMIT[2] (µg/kg)
POLYCHLORINATED BIPHENYLS (PCBs)							
Total PCBs	1336363	4,000	-	28,000,000	330	20	NA[6]
Aroclor-1016	12674112		Part 201 N/A		-	10	NA[6]
Aroclor-1221	11104282		Part 201 N/A		-	20	NA[6]
Aroclor-1232	11141165		Part 201 N/A		-	10	NA[6]
Aroclor-1242	53469219		Part 201 N/A		-	10	NA[6]
Aroclor-1248	12672296		Part 201 N/A		-	10	NA[6]
Aroclor-1254	11097691		Part 201 N/A		-	10	NA[6]
Aroclor-1260	11096825		Part 201 N/A		-	10	NA[6]
VOLATILE ORGANIC COMPOUNDS							
1,1,1-Trichloroethane	71556	4,000	-	67,000,000,000	50	50.00	11.10
1,1,2,2-Tetrachloroethane	79345	64	-	68,000,000	50	50.00	13.80
1,1,2-Trichloroethane	79005	100	-	250,000,000	50	50.00	10.40
1,1-Dichloroethane	75343	15,000	-	33,000,000,000	50	50.00	9.06
1,1-Dichloroethylene	75354	62	-	78,000,000	50	50.00	19.80
1,2,4-Trichlorobenzene	120821	1,800	-	25,000,000,000	330	200.00	21.80
1,2-Dibromoethane (ethylene dibromide)	106934	20	-	18,000,000	20	200.00	0.73
1,2-Dichlorobenzene	95501	360	-	100,000,000,000	100	50.00	8.47
1,2-Dichloroethane	107062	100	-	150,000,000	50	50.00	11.40
1,2-Dichloropropane	78875	100	-	270,000,000	50	50.00	19.00
1,3-Dichlorobenzene	541731	170	-	170,000	100	50.00	10.20
1,4-Dichlorobenzene	106467	290	-	570,000,000	100	50.00	8.69
2-Butanone (MEK)	78933	44,000	-	67,000,000,000	750	2,000.00	322.00
2-Hexanone	591786	20,000	-	2,700,000,000	2,500	2,000.00	396.00
4-Methyl-2-pentanone (MIBK)	108101	36,000	-	140,000,000,000	2,500	2,000.00	340.00
Acetone	67641	15,000	-	390,000,000,000	1,000	2,000.00	240.00
Benzene	71432	100	-	470,000,000	50	50.00	10.50
Bromodichloromethane	75274	1,200	-	110,000,000	100	50.00	8.70

QAPP Worksheet #15-2
Reference Limits and Evaluation (Soil/Sediment)

PARAMETER[1][4]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µg/kg)			PART 201 TDLs (µg/kg)	QUANTITATION LIMIT[2] (µg/kg)	METHOD DETECTION LIMIT[2] (µg/kg)
Bromoform	75252	1,600	-	3,600,000,000	100	50.00	27.90
Bromomethane	74839	200	-	330,000,000	200	50.00	21.70
Carbon disulfide	75150	16,000	-	47,000,000,000	250	50.00	15.90
Carbon tetrachloride	56235	100	-	170,000,000	50	50.00	13.00
Chlorobenzene	108907	940	-	4,700,000,000	50	50.00	9.33
Chloroethane	75003	8,600	-	670,000,000,000	250	50.00	17.30
Chloroform	67663	1,500	-	1,600,000,000	50	50.00	9.58
Chloromethane	74873	2,300	-	4,900,000,000	250	50.00	13.60
cis-1,2-Dichloroethylene	156592	1,400	-	2,300,000,000	50	50.00	11.60
cis-1,3-Dichloropropene	542756	170	-	780,000,000	100	50.00	8.90
Dibromochloromethane	124481	1,600	-	160,000,000	100	50.00	8.17
Dibromochloropropane	96128	10	-	13,000,000	10	200.00	99.10
Dichlorodifluoromethane	75718	95,000	-	3,300,000,000,000	250	50.00	16.60
Ethylbenzene	100414	360	-	13,000,000,000	50	50.00	9.74
Isopropyl benzene	98828	91,000	-	5,800,000,000	250	200.00	9.30
Methylene chloride	75092	100	-	8,300,000,000	100	200.00	19.30
Methyl-tert-butyl ether (MTBE)	1634044	800	-	200,000,000,000	250	50.00	15.00
Styrene	100425	2,200	-	6,900,000,000	50	50.00	9.43
Tetrachloroethylene	127184	100	-	6,800,000,000	50	50.00	14.00
Toluene	108883	2,800	-	27,000,000,000	100	50.00	9.75
trans-1,2-Dichloroethylene	156605	2,000	-	4,700,000,000	50	50.00	13.90
trans-1,3-Dichloropropene	542756	170	-	780,000,000	100	50.00	9.10
Trichloroethylene	79016	100	-	2,300,000,000	50	50.00	18.80
Trichlorofluoromethane	75694	52,000	-	3,800,000,000,000	100	50.00	17.00
Vinyl chloride	75014	40	-	890,000,000	40	50.00	21.10
m,p-Xylenes	1330207	700	-	290,000,000,000	150	50.00	18.60
o-Xylenes	1330207	700	-	290,000,000,000	150	50.00	7.85

QAPP Worksheet #15-2
Reference Limits and Evaluation (Soil/Sediment)

PARAMETER[1][4]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µg/kg)			PART 201 TDLs (µg/kg)	QUANTITATION LIMIT[2] (µg/kg)	METHOD DETECTION LIMIT[2] (µg/kg)
SEMI VOLATILE ORGANIC COMPOUNDS							
2,4,5-Trichlorophenol	95954	39,000	-	23,000,000,000	330	330	20
2,4,6-Trichlorophenol	88062	330	-	1,300,000,000	330	330	13
2,4-Dichlorophenol	120832	380	-	5,100,000,000	330	330	20
2,4-Dimethylphenol	105679	7,400	-	4,700,000,000	330	330	63
2,4-Dinitrotoluene	121142	430	-	20,000,000	330	330	27
2-Chlorophenol	95578	440	-	19,000,000	330	330	20
2-Methylnaphthalene	91576	57,000	-	37,000,000	330	330	30
2-Methylphenols (J)	1319773	1,400	-	6,700,000,000	330	330	23
2-Nitrophenol	88755	400	-	2,900,000	330	330	20
3,3-Dichlorobenzidine	91941	2,000	-	8,200,000	2,000	330	100
4,6-Dinitro-2-methylphenol (2-methyl-4,6-dinitrophenol)	534521	830	-	360,000	830	330	20
4-Chloro-3-methylphenol	59507	280	-	20,000,000	280	330	20
4-Methylphenol (J)	1319773	1,400	-	6,700,000,000	330	330	23
Acenaphthene	83329	4,400	-	14,000,000,000	330	330	20
Acenaphthylene	208968	5,900	-	2,300,000,000	330	330	17
Anthracene	120127	41,000	-	67,000,000,000	330	330	17
Benzo(a)anthracene	56553	20,000	-	160,000	330	330	10
Benzo(a)pyrene	50328	2,000	-	1,900,000	330	330	13
Benzo(b)fluoranthene	205992	20,000	-	160,000	330	330	17
Benzo(g,h,i)perylene	191242	2,500,000	-	800,000,000	330	330	7
Benzo(k)fluoranthene	207089	200,000	-	1,600,000	330	330	20
bis(2-Chloroethyl)ether (I)	111444	100	-	12,000,000	100	330	37
bis(2-Ethylhexyl)phthalate	117817	2,800,000	-	890,000,000	330	330	30
Butyl benzyl phthalate	85687	13,000	-	47,000,000,000	330	330	23
Carbazole	86748	1,100	-	3,400,000	330	330	40
Chrysene	218019	2,000,000	-	16,000,000	330	330	13

QAPP Worksheet #15-2
Reference Limits and Evaluation (Soil/Sediment)

PARAMETER[1][4]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µg/kg)			PART 201 TDLs (µg/kg)	QUANTITATION LIMIT[2] (µg/kg)	METHOD DETECTION LIMIT[2] (µg/kg)
Dibenzo(a,h)anthracene	53703	2,000	-	16,000	330	330	7
Dibenzofuran	132649		1,700		330	330	23
Diethyl phthalate	84662	2,200	-	3,300,000,000	330	330	17
Dimethyl phthalate	131113	790,000	-	3,300,000,000	330	330	17
Di-n-butylphthalate	84742	11,000	-	3,300,000,000	330	330	10
Di-n-octyl phthalate	117840	6,900,000	-	140,000,000	330	330	13
Fluoranthene	206440	5,500	-	9,300,000,000	330	330	27
Fluorene	86737	5,300	-	9,300,000,000	330	330	30
Hexachlorobenzene (C-66)	118741	350	-	8,500,000	330	330	23
Hexachlorobutadiene (C-46)	87683	91	-	180,000,000	50	330	40
Hexachlorocyclopentadiene (C-56)	77474	30,000	-	13,000,000	330	330	330
Hexachloroethane	67721	310	-	230,000,000	300	330	33
Indeno(1,2,3-cd)pyrene	193395	20,000	-	160,000	330	330	7
Isophorone	78591	6,200	-	12,000,000,000	330	330	17
Naphthalene	91203	870	-	200,000,000	330	330	27
Nitrobenzene (I)	98953	330	-	47,000,000	330	330	37
n-Nitroso-di-n-propylamine	621647	330	-	2,000,000	330	330	23
N-Nitrosodiphenylamine	86306	5,400	-	11,000,000	330	330	53
Pentachlorophenol	87865	22	-	130,000,000	20	330	30
Phenanthrene	85018	5,300	-	7,200,000	330	330	17
Phenol	108952	4,200	-	40,000,000,000	330	330	23
Pyrene	129000	480,000	-	6,700,000,000	330	330	40

QAPP Worksheet #15-2
Reference Limits and Evaluation (Soil/Sediment)

PARAMETER[1][4]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µg/kg)	PART 201 TDLs (µg/kg)	QUANTITATION LIMIT[2] (µg/kg)	METHOD DETECTION LIMIT[2] (µg/kg)

Notes:

[1] The parameters included in this table are those included in the USEPA Contract Laboratory Program (CLP) Target Compound List (TCL) and Target Analyte List (TAL), except those that are not on the Part 201 list: Calcium, Potassium, Chromium IV, 1,2,3-Trichlorobenzene, Bromochloromethane, Cyclohexane, Methyl Acetate, Methylcyclohexane (methyl phenol), 1,1-Biphenyl, 2,2-oxybis(1-Chloropropane), 2,4-Dinitrophenol, 2,6-Dinitrotoluene, 2-Nitroaniline, 3-Nitroaniline, 4-Bromophenyl-phenylether, 4-Chloroaniline, 4-Chlorophenyl-phenylether, 4-Nitroaniline, 4-Nitrophenol, Benzaldehyde, bis(2-Chloroethoxy)methane.

Or those that are not typically analyzed by WATS or CAS: 1,1,2-Trichloro-1,2,2-triflouroethane, 1,2,4,5-Tetrachlorobenzene, Acetophenone, Atrazine, beta-Chloronaphthalene, Caprolactum.

[2] The actual Quantitation Limits and Method Detection Limits will vary with sample size, for the analysis of PCBs and PAHs.

[3] Potentially applicable Michigan Part 201 Criteria are from the Michigan Department of Environmental Quality (MDEQ) Remediation and Redevelopment Division (RRD) Operational Memorandum No. 1, dated December 10, 2004.

[4] VOC analysis will be conducted by Columbia Analytical Services. All other analyses will be conducted by Weyerhaeuser Analytical Testing Services

[5] MDLs for these parameters are only calculated for water samples.

[6] The MDL actually achievable in a given analysis will vary depending on detector response characteristics, irreducible noise from instrument electronics, and matrix effects.

WATS = Weyerhaeuser Analytical Testing Service.

NA = Not Available

NS = no standard.

QAPP Worksheet #15-3
Reference Limits and Evaluation (Groundwater)

PARAMETER[1]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µG/L)			PART 201 TARGET DETECTION LIMIT (µG/L)	AQUEOUS QUANTITATION LIMIT[2] (µG/L)	METHOD DETECTION LIMIT[2] (µG/L)
METALS AND CYANIDE							
Aluminum	7429905	50	-	64,000,000	50	5	0.3
Antimony	7440360	2	-	68,000	2	0.5	0.07
Arsenic	7440382	10	-	4,300	5	3	0.03
Barium	7440393	1159.40	-	14,000,000	100	5	0.02
Beryllium	7440417	4	-	290,000	1	0.5	0.02
Cadmium	7440439	2.5	-	190,000	1	0.5	0.01
Chromium (III)	16065831	100	-	290,000,000	10	10	0.03
Chromium (VI)	18540299	11	-	460,000	10	10	0.03
Cobalt	7440484	40	-	2,400,000	20	10	0.008
Copper	7440508	19.59	-	7,400,000	4	0.5	0.03
Iron	7439896	300	-	58,000,000	200	50	0.7
Lead	7439921	4	-	27	3	3	0.006
Magnesium	7439954	400,000	-	1,000,000,000	1,000	100	0.2
Manganese	7439965	50	-	9,100,000	50	5	0.02
Mercury (total)[4]	7439976	0.0013	-	56	0.001	1 (ng/L)	0.06 (ng/L)
Nickel	7440020	100.00	-	74,000,000	20	50	0.06
Selenium	7782492	5	-	970,000	5	3	0.1
Silver	7440224	0.2	-	1,500,000	0.2	0.5	0.04
Sodium	17341252	120,000	-	1,000,000,000	1,000	500	1
Thallium	7440280	2	-	13,000	2	3	0.007
Vanadium	7440622	4.5	-	970,000	4	0.5	0.2
Zinc	7440666	256.78	-	110,000,000	50	10	0.3
Cyanide	57125	5.2	-	57,000	5	9	3

QAPP Worksheet #15-3
Reference Limits and Evaluation (Groundwater)

PARAMETER[1]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µG/L)			PART 201 TARGET DETECTION LIMIT (µG/L)	AQUEOUS QUANTITATION LIMIT[2] (µG/L)	METHOD DETECTION LIMIT[2] (µG/L)
POLYCHLORINATED BIPHENYLS (PCBs)							
Total PCBs	1336363	0.2	-	45	0.2	0.2	NA[5]
Aroclor-1016	12674112		Part 201 N/A			0.2	NA[5]
Aroclor-1221	11104282		Part 201 N/A			0.2	NA[5]
Aroclor-1232	11141165		Part 201 N/A			0.2	NA[5]
Aroclor-1242	53469219		Part 201 N/A			0.2	NA[5]
Aroclor-1248	12672296		Part 201 N/A			0.2	NA[5]
Aroclor-1254	11097691		Part 201 N/A			0.2	NA[5]
Aroclor-1260	11096825		Part 201 N/A			0.2	NA[5]
VOLATILE ORGANIC COMPOUNDS							
1,1,1-Trichloroethane	71556	200	-	1,300,000	1	0.5	0.13
1,1,2,2-Tetrachloroethane	79345	3.2	-	77,000	1	0.5	0.09
1,1,2-Trichloroethane	79005	5	-	110,000	1	0.5	0.10
1,1-Dichloroethane	75343	740	-	2,400,000	1	0.5	0.09
1,1-Dichloroethylene	75354	7	-	11,000	1	0.5	0.08
1,2,4-Trichlorobenzene	120821	30	-	300,000	5	0.5	0.07
1,2-Dibromoethane (ethylene dibromide)	106934	0.05	-	15,000	0.05	0.5	0.11
1,2-Dichlorobenzene	95501	16	-	160,000	1	0.5	0.06
1,2-Dichloroethane	107062	5	-	59,000	1	0.5	0.12
1,2-Dichloropropane	78875	5	-	36,000	1	0.5	0.09
1,3-Dichlorobenzene	541731	6.6	-	2,000	1	0.5	0.09
1,4-Dichlorobenzene	106467	13	-	74,000	1	0.5	0.11
2-Butanone (MEK)	78933	2,200	-	240,000,000	25	5	0.92
2-Hexanone	591786	1,000	-	8,700,000	50	5	0.84
4-Methyl-2-pentanone (MIBK)	108101	1,800	-	20,000,000	50	5	0.77
Acetone	67641	730	-	1,000,000,000	50	5	0.74
Benzene	71432	5	-	35,000	1	0.5	0.10
Bromodichloromethane	75274	80	-	37,000	1	0.5	0.08

QAPP Worksheet #15-3
Reference Limits and Evaluation (Groundwater)

PARAMETER[1]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µG/L)			PART 201 TARGET DETECTION LIMIT (µG/L)	AQUEOUS QUANTITATION LIMIT[2] (µG/L)	METHOD DETECTION LIMIT[2] (µG/L)
Bromoform	75252	80	-	3,100,000	1	0.5	0.09
Bromomethane	74839	10	-	70,000	5	0.5	0.21
Carbon disulfide	75150	800	-	1,200,000	5	0.5	0.09
Carbon tetrachloride	56235	5	-	4,600	1	0.5	0.09
Chlorobenzene	108907	47	-	470,000	1	0.5	0.08
Chloroethane	75003	430	-	5,700,000	5	0.5	0.09
Chloroform	67663	77	-	180,000	1	0.5	0.07
Chloromethane	74873	260	-	490,000	5	0.5	0.15
cis-1,2-Dichloroethylene	156592	70	-	210,000	1	0.5	0.09
cis-1,3-Dichloropropene	542756	8.5	-	26,000	1	0.5	0.16
Dibromochloromethane	124481	80	-	110,000	5	0.5	0.09
Dibromochloropropane	96128	0.2	-	1,200	0.2	0.5	0.26
Dichlorodifluoromethane	75718	1,700	-	300,000	5	0.5	0.12
Ethylbenzene	100414	18	-	170,000	1	0.5	0.10
Isopropyl benzene	98828	800	-	56,000	5	0.5	0.15
Methylene chloride	75092	5	-	1,400,000	5	0.5	0.04
Methyl-tert-butyl ether (MTBE)	1634044	40	-	47,000,000	5	0.5	0.12
Styrene	100425	80	-	310,000	1	0.5	0.07
Tetrachloroethylene	127184	5	-	170,000	1	0.5	0.12
Toluene	108883	140	-	530,000	1	0.5	0.09
trans-1,2-Dichloroethylene	156605	100	-	220,000	1	0.5	0.11
trans-1,3-Dichloropropene	542756	8.5	-	26,000	1	0.5	0.15
Trichloroethylene	79016	5	-	97,000	1	0.5	0.10
Trichlorofluoromethane	75694	2,600	-	1,100,000	1	0.5	0.08
Vinyl chloride	75014	2	-	13,000	1	0.5	0.06
m,p-Xylene	1330207	35	-	190,000	3	0.5	0.27
o-Xylene	1330207	35	-	190,000	3	0.5	0.12

QAPP Worksheet #15-3
Reference Limits and Evaluation (Groundwater)

PARAMETER[1]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µG/L)			PART 201 TARGET DETECTION LIMIT (µG/L)	AQUEOUS QUANTITATION LIMIT[2] (µG/L)	METHOD DETECTION LIMIT[2] (µG/L)
SEMI VOLATILE ORGANIC COMPOUNDS							
2,4,5-Trichlorophenol	95954	730	-	170,000	5	10	0.6
2,4,6-Trichlorophenol	88062	4.4	-	10,000	4	10	0.4
2,4-Dichlorophenol	120832	19	-	48,000	10	10	0.6
2,4-Dimethylphenol	105679	370	-	520,000	5	10	1.9
2,4-Dinitrotoluene	121142	7.7	-	8,600	5	10	0.8
2-Chlorophenol	95578	22	-	94,000	10	10	0.6
2-Methylnaphthalene	91576	260	-	25,000	5	10	0.9
2-Methylphenol (J)	1319773	71	-	810,000	10	10	0.7
2-Nitrophenol	88755	20	-	79,000	5	10	0.6
3,3-Dichlorobenzidine	91941	0.3	-	180	0.3	10	3
4,6-Dinitro-2-methylphenol (2-methyl-4,6-dinitrophenol)	534521	20	-	9,500	20	10	0.6
4-Chloro-3-methylphenol	59507	7.4	-	79,000	5	10	0.6
4-Methylphenol (J)	1319773	71	-	810,000	10	10	0.7
Acenaphthene	83329	19	-	4,200	5	10	0.6
Acenaphthylene	208968	52	-	3,900	5	10	0.5
Anthracene	120127		43		5	10	0.5
Benzo(a)anthracene	56553	2.1	-	9	1	10	0.3
Benzo(a)pyrene	50328	1	-	5	1	10	0.4
Benzo(b)fluoranthene	205992		1.5		1	10	0.5
Benzo(g,h,i)perylene	191242		1		1	10	0.2
Benzo(k)fluoranthene	207089		1		1	10	0.6
bis(2-Chloroethyl)ether (I)	111444	1	-	210,000	1	10	1.1
bis(2-Ethylhexyl)phthalate	117817	6	-	320	5	10	0.9
Butyl benzyl phthalate	85687	6.9	-	2,700	5	10	0.7
Carbazole	86748	10	-	7,400	10	10	1.2
Chrysene	218019		1.6		1	10	0.4

QAPP Worksheet #15-3
Reference Limits and Evaluation (Groundwater)

PARAMETER[1]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µG/L)			PART 201 TARGET DETECTION LIMIT (µG/L)	AQUEOUS QUANTITATION LIMIT[2] (µG/L)	METHOD DETECTION LIMIT[2] (µG/L)
Dibenzo(a,h)anthracene	53703		2		2	10	0.2
Dibenzofuran	132649		4		4	10	0.7
Diethyl phthalate	84662	110	-	1,100,000	5	10	0.5
Dimethyl phthalate	131113	73,000	-	4,200,000	5	10	0.5
Di-n-butylphthalate	84742	9.7	-	11,000	5	10	0.3
Di-n-octyl phthalate	117840	130	-	400	5	10	0.4
Fluoranthene	206440	1.6	-	210	1	10	0.8
Fluorene	86737	12	-	2,000	5	10	0.9
Hexachlorobenzene (C-66)	118741	0.2	-	3,000	0.2	10	0.7
Hexachlorobutadiene (C-46)	87683	0.05	-	3,200	0.05	10	1.2
Hexachlorocyclopentadiene (C-56)	77474	50	-	1,600	5	10	10
Hexachloroethane	67721	5.3	-	50,000	5	10	1
Indeno(1,2,3-cd)pyrene	193395		2		2	10	0.2
Isophorone	78591	310	-	990,000	5	10	0.5
Naphthalene	91203	13	-	31,000	5	10	0.8
Nitrobenzene (I)	98953	3.4	-	550,000	3	10	1.1
n-Nitroso-di-n-propylamine	621647	5	-	360	5	10	0.7
N-Nitrosodiphenylamine	86306	270	-	35,000	5	10	1.6
Pentachlorophenol	87865	1	-	200	1	10	0.9
Phenanthrene	85018	2.4	-	1,000	2	10	0.5
Phenol	108952	210	-	29,000,000	5	10	0.7
Pyrene	129000		140		5	10	1.2

QAPP Worksheet #15-3
Reference Limits and Evaluation (Groundwater)

PARAMETER[1]	CHEMICAL ABSTRACT SERVICE NUMBER	RANGE OF POTENTIALLY APPLICABLE PART 201 CRITERIA[3] (µG/L)	PART 201 TARGET DETECTION LIMIT (µG/L)	AQUEOUS QUANTITATION LIMIT[2] (µG/L)	METHOD DETECTION LIMIT[2] (µG/L)

Notes:

[1] The parameters included in this table are those included in the USEPA Contract Laboratory Program (CLP) Target Compound List (TCL) and Target Analyte List (TAL), except those that are not on the Part 201 list: Calcium, Potassium, Chromium IV, 1,2,3-Trichlorobenzene, Bromochloromethane, Cyclohexane, Methyl Acetate, Methylcyclohexane (methyl phenol), 1,1-Biphenyl, 2,2-oxybis(1-Chloropropane), 2,4-Dinitrophenol, 2,6-Dinitrotoluene, 2-Nitroaniline, 3-Nitroaniline, 4-Bromophenyl-phenylether, 4-Chloroaniline, 4-Chlorophenyl-phenylether, 4-Nitroaniline, 4-Nitrophenol, Benzaldehyde, bis(2-Chloroethoxy)methane.

Or those that are not typically analyzed by WATS or CAS: 1,1,2-Trichloro-1,2,2-triflouroethane, 1,2,4,5-Tetrachlorobenzene, Acetophenone, Atrazine, beta-Chloronaphthalene, Caprolactum.

[2] The actual Quantitation Limits and Method Detection Limits will vary with sample size, for the analysis of PCBs and PAHs.

[3] Potentially applicable Michigan Part 201 Criteria are from the Michigan Department of Environmental Quality (MDEQ) Remediation and Redevelopment Division (RRD) Operational Memorandum No. 1, dated December 10, 2004.

[4] VOC analysis will be conducted by Columbia Analytical Services. All other analyses will be conducted by Weyerhaeuser Analytical Testing Services

[5] The MDL actually achievable in a given analysis will vary depending on detector response characteristics, irreducible noise from instrument electronics, and matrix effects.

WATS = Weyerhaeuser Analytical Testing Service.

NA = Not Available

NS = no standard.

QAPP Worksheet #15-4
Reference Limits and Evaluation (Free Product)

ANALYTE	CAS NUMBER	PROJECT ACTION LIMIT[1]	FREE PRODUCT (µg/L)[2]	
			LABORATORY MDL	LABORATORY RL
PCBs (U.S. EPA 8082)				
Aroclor - 1016	12674-11-2	--	0.20	0.2
Aroclor - 1221	11104-28-2	--	--	0.2
Aroclor - 1232	11141-16-5	--	0.30	0.2
Aroclor - 1242	53469-21-9	--	0.24	0.2
Aroclor - 1248	12672-29-6	--	0.33	0.2
Aroclor - 1254	11097-69-1	--	0.25	0.2
Aroclor - 1260	11096-82-5	--	0.24	0.2
Total PCBs	1336-36-3	0.2 µg/L	--	0.2
Physical Properties				
Viscosity (ASTM D445)	--	--	--	--
Specific Gravity (ASTM D287)	--	--	--	--

Footnotes:
(1) Standards are consistent with the Multi-Area Quality Assurance Project Plan (Rev. 00) For the Kalamazoo River Study Group (Arcadis BBL, 2007b), which are from the MDEQ's Substantive Requirements Document for the Plainwell Dam Superfund Site, dated February 13, 2007. The TSS standards are on a monthly and daily basis, respectively.
(2) Concentrations are in µg/L, except where noted.

Notes:
RL = reporting limit.
MDL = Method Detection Limit.
µg/L = micrograms per liter.
mg/L = milligrams per liter.
PCBs = polychlorinated biphenyls.

QAPP Worksheet #16-1
Project Schedule/Timeline
(Emergency Response Activities in the Former Powerhouse Discharge Channel)

ACTIVITIES	ORGANIZATION	DATES (MM/DD/YY)		DELIVERABLE	DELIVERABLE DUE DATE
		ANTICIPATED DATE(S) OF INITIATION	ANTICIPATED DATE OF COMPLETION		
Geotechnical Investigation	RMT	5/21/07	6/30/07	Technical Memorandum	Incorporated into Design Report
Bathymetry and visual sediment assessment	RMT	6/12/07	6/22/07	Technical Memorandum	Incorporated into Design Report
Remedial design	RMT	6/4/07	6/25/07	Emergency Response Work Plan	6/26/7 – Draft 8/1/07 – Final
		6/1/07	6/25/07	Field Sampling Plan	6/26/07 – Draft 8/1/07 – Final
		5/15/07	6/20/07	Quality Assurance Project Plan (QAPP)	6/21/07 – Draft 8/1/07 – Final
		5/30/07	7/27/07	Emergency Response Design Plan, including Schedule	7/28/07
Remedial construction	RMT	7/30/07	11/8/07	Emergency Response Documentation Report	1/31/08

QAPP Worksheet #16-2
Project Schedule/Timeline
(Emergency Response Activities on the Plainwell Mill Banks)

ACTIVITIES	ORGANIZATION	DATES (MM/DD/YY)		DELIVERABLE	DELIVERABLE DUE DATE
		ANTICIPATED DATE(S) OF INITIATION	ANTICIPATED DATE OF COMPLETION		
Visual soils and sediment assessment	RMT	7/16/07	7/30/07	Technical Memorandum	Incorporated into Design Report
Remedial design	RMT	7/23/07	6/25/07	Emergency Action Conceptual Design Approach	8/14/07
		8/15/07	9/30/07	Field Sampling Plan Addendum	9/10/07 – Draft 9/30/07 – Final
		8/15/07	9/30/07	Quality Assurance Project Plan (QAPP) Addendum	9/10/07 – Draft 9/30/07 – Final
		8/15/07	9/30/07	Emergency Response Design Plan, including Schedule	9/5/07
Remedial construction	RMT	9/15/07	12/31/07 (field work) 3/30/08 (report)	Emergency Response Documentation Report	2/28/08

QAPP Worksheet #16-3
Project Schedule/Timeline
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)

ACTIVITIES	ORGANIZATION	DATES (MM/DD/YY)		DELIVERABLE	DELIVERABLE DUE DATE
		ANTICIPATED DATE(S) OF INITIATION	ANTICIPATED DATE OF COMPLETION		
Conduct predesign field activities	Weyerhaeuser/RMT	June 2008, as weather allows	June 2008	Section of the RD report or a technical memorandum	To be determined pending U.S. EPA approval of accelerated RD process
Preliminary Design	Weyerhaeuser/RMT	June 2008	September 2008	Preliminary Design Report	
Pre-Final Design	Weyerhaeuser/RMT	October 2008	November 2008	Pre-Final Design Report	
Final Design	Weyerhaeuser/RMT	December 2008	January 2009	Final Design Report	
Draft Remedial Action Workplan	Weyerhaeuser/RMT	January 2009	February 2009	Draft Remedial Action Workplan	
Final Remedial Action Workplan	Weyerhaeuser/RMT	April 2009	May 2009	Final Remedial Action Workplan	

QAPP Worksheet #16-4
Project Schedule/Timeline
(Phase 2 of Addendum No. 1 - Test Pits & Plainwell Mill RI/FS – Phase I Groundwater Assessment)

ACTIVITIES	ORGANIZATION	DATES (MM/DD/YY)		DELIVERABLE	DELIVERABLE DUE DATE
		ANTICIPATED DATE(S) OF INITIATION	ANTICIPATED DATE OF COMPLETION		
Phase 2 of Addendum No. 1	RMT	9/1/08	1/1/09	Technical Memorandum	60 Days After Completion of Field Activities or Receipt of Data
Plainwell Mill RI/FS – Phase I Groundwater Assessment & Coal Tunnel Activities	RMT	9/1/08	1/1/09	Summary of Results in Appropriate Monthly Progress Report	1st Monthly Progress Report – 60 Days After Completion of Field Activities or Receipt of Data

QAPP Worksheet #17-1
Sampling Design and Rationale (Former Powerhouse Discharge Channel)

Describe and provide a rationale for choosing the sampling approach (*e.g.*, grid system, biased statistical approach):

Surface Water Quality:

The upstream turbidity samples will be taken at a location near the Plainwell spillway and then at two downstream locations approximately 200 feet and 300 feet downstream of the work area along the path of the water flowing past the work area as described in worksheet #11. The sample collected at 200 feet downstream will serve as a warning to address site actions if elevated results are measured. Water quality may be impacted in the immediate vicinity of the former powerhouse discharge channel; therefore, the sampling locations were established downstream in the main channel to determine if the residuals removal activities were impacting water quality of the wider river system.

Sediment Sampling:

The suggested sampling protocol for the former powerhouse discharge channel is consistent with the grid format and the quantity of samples targeted for confirmation sampling for the Plainwell Impoundment TCRA. Sampling of the former powerhouse discharge channel would consist of establishing sample areas that will each contain approximately 20 grids with each grid having an area of approximately 400 square feet. Five samples will be collected from each area in a semi-random fashion that will ensure adequate coverage across the entire area. All confirmatory samples will be collected by hand (*e.g.* using a trowel), targeting a sediment depth of 0-6 inches.

Water Treatment System:

Sampling for the water treatment system is similar to that proposed for the Plainwell Impoundment TCRA. This sampling program will provide adequate protection for the receiving surface water.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations):

Refer to Worksheet #18-1.

QAPP Worksheet #17-2
Sampling Design and Rationale (Plainwell Mill Banks)

Describe and provide a rationale for choosing the sampling approach (*e.g.*, grid system, biased statistical approach):

Surface Water Quality:

Same as Worksheet 17-1.

Sediment Sampling:

The suggested sampling protocol for the Plainwell Mill banks is consistent with the grid format and the quantity of samples targeted for confirmation sampling for the Plainwell Impoundment TCRA. Sampling of the Plainwell Mill banks would consist of establishing sample areas that will each contain approximately 20 grids; for floodplain/near-shore sediment excavation areas, the sample area will cover approximately 5,000 square feet and will be divided into 20 grids (10 feet by 25 feet); for bank excavation areas, the sample area will cover 1,000 square feet and will be divided into 20 grids (5 feet by 10 feet) . Five samples will be collected from each area in a semi-random fashion that will ensure adequate coverage across the entire area. All confirmatory samples will be collected by hand (*e.g.* using a trowel), targeting a soil and sediment depth of 0 to 6 inches.

Water Treatment System:

Same as Worksheet 17-1.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations):

Refer to Worksheet #18-1.

QAPP Worksheet #17-3
Sampling Design and Rationale
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)

Describe and provide a rationale for choosing the sampling approach (*e.g.*, grid system, biased statistical approach):

The sampling approach was based on a review and determination of potential additional data needs. Specifically, RMT reviewed the available information, which was gathered during numerous site investigations (see Worksheet #13-1), and then compared the scope of the available information to that which may be needed to complete the remedial design. The sampling approach detailed in Worksheets #10-3 and #11-3 (i.e., test pits to visually determine the extent of paper residuals beyond the toe of the landfill, Geoprobe® borings to determine the thickness of paper residuals along the property lines, and gas measurements in existing monitoring wells and the Geoprobe® borings) will be used to supplement the already extensive information that is available for the remedial design.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations):

No samples will be collected for laboratory analysis.

QAPP Worksheet #17-4
Sampling Design and Rationale
(Phase 2 of Addendum No. 1 - Test Pits & Plainwell Mill RI/FS – Phase I Groundwater Assessment)

Describe and provide a rationale for choosing the sampling approach (*e.g.*, grid system, biased statistical approach):

Phase 2 of Addendum No. 1 - Test Pits:

A biased sampling approach will be employed for the test pit investigation. The test pit locations were selected based upon previous site analytical data, site observations, a geophysical survey in the area of interest, and a historical review that identified potential outfalls and historical storage locations. In addition, selection of samples for analysis based upon a number of site observations including visible residuals, petroleum impacts, PID readings, notable odors, and soil stratigraphy.

Plainwell Mill RI/FS – Initial Groundwater Assessment:

Presently a limited amount of groundwater data has been collected at the site through the use of temporary monitoring wells. Data collected from temporary monitoring wells is not appropriate for comparison to ARARs and do not provide information regarding the groundwater gradient or elevation at the site. Therefore, the proposed monitoring well network for the initial groundwater assessment will be biased to provide data down gradient from areas of known or potential contamination and will be situated to provide valuable data regarding subsurface water elevations.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations):

Refer to Worksheet #18-1.

QAPP Worksheet #18-1
Sampling Locations and Methods/SOP Requirements
(Emergency Response Activities)

SAMPLING LOCATION/ID NUMBER	MATRIX	ANALYTICAL GROUP	CONCENTRATION LEVEL	NUMBER OF SAMPLES[2] (identify field duplicates)	SAMPLING SOP REFERENCE[1]	RATIONALE FOR SAMPLING LOCATIONS
Surface water quality monitoring	Water	Total PCBs	Normal	Weekly during construction	F-1	One upstream and two downstream locations (mid-channel, mid-depth)
		Turbidity	Normal	Periodic monitoring during construction using in-stream sampling equipment	F-2	One upstream and two downstream locations (mid-channel, mid-depth)
Post-construction in former powerhouse discharge channel	Sediment	Total PCBs	Normal	Minimum of five samples per 8,000-square foot area, as described in Worksheet #17	F-3	Surface samples (*i.e.*, 0 to 6 inches) obtained to verify removal effectiveness
Water treatment system	Treated water	Total PCB TSS Total phosphorus	Normal	Twice per week during system operation	F-5	Discharge location

Footnotes:

(1) See Project Sampling SOP References table (Worksheet #21).

(2) Field duplicates will be collected at a frequency of one duplicate for every 10 samples (Worksheet #20).

QAPP Worksheet #18-2
Sampling Locations and Methods/SOP Requirements
(Predesign Investigations for the Remedial Design for the 12th Street Landfill)

SAMPLING LOCATION/ID NUMBER	MATRIX	ANALYTICAL GROUP	CONCENTRATION LEVEL	NUMBER OF SAMPLES (identify field duplicates)	SAMPLING SOP REFERENCE[1]	RATIONALE FOR SAMPLING LOCATIONS
Test pits	Soil	None	Not applicable	3 test pits in the wetlands north of the landfill, 3 test pits on the asphalt plant property, and 4 test pits on the State property[2]	Not applicable	Potential data gaps, based on previously available information (see Worksheet #13-1)
Geoprobe® borings	Soil	None	Not applicable	9 Geoprobe® borings into the 12th Street Landfill At least 2 Geoprobe® borings near the southern end of the landfill	F-5	Potential data gaps, based on previously available information (see Worksheet #13-1)
Landfill gas measurements	Soil gas	None	Not applicable	As may be readily accessible, 3 existing groundwater monitoring wells screened in the vadose zone (MW-6A, MW-7A, and MW-8A) and Geoprobe® borings installed as part of this investigation	F-7	May be useful to support existing information (see Worksheet #13-1). Collection of this data is not essential because Weyerhaeuser has decided to include a gas venting system in the final landfill cover design.

Footnotes:

(1) See Project Sampling SOP References table (Worksheet #21).

(2) In the event that in-field conditions limit the use of test pit excavating equipment (e.g., a backhoe), other tools, such as hand augers or shovels, may be used instead. In such instances, the U.S. EPA project manager, or designated alternate, will be contacted to discuss the situation and to agree on a course of action.

QAPP Worksheet #18-3
Sampling Locations and Methods/SOP Requirements
(Phase 2 of Addendum No. 1 - Test Pits & Plainwell Mill RI/FS – Phase I Groundwater Assessment)

SAMPLING LOCATION/ID NUMBER	MATRIX	ANALYTICAL GROUP	CONCENTRATION LEVEL	NUMBER OF SAMPLES (identify field duplicates)	SAMPLING SOP REFERENCE[1]	RATIONALE FOR SAMPLING LOCATIONS
Test Pit Sampling in the Oil Impacted Storage Area	Soil	PCBs, total petroleum hydrocarbons (DRO/GRO)	Normal	Approximately 10 test pits will be excavated and a minimum of 1 soil sample per test pit will be collected[2], plus 1 field duplicate sample	F-7/F-8	It is estimated that 10 test pits will be excavated. A minimum of one soil sample will be collected from each test pit.
Test Pit Sampling in the Elevated PCB Area	Soil	PCBs, total petroleum hydrocarbons (DRO/GRO)	Normal	Approximately 5 test pits will be excavated and a minimum of 1 soil sample per test pit will be collected[2], plus 1 field duplicate sample	F-7/F-8	It is estimated that 5 test pits will be excavated. A minimum of one soil sample will be collected from each test pit.
Additional Samples From Test Pits	Soil	PAHs, VOCs and/or RCRA metals	Normal	Up to 5 samples with elevate PID readings or the presence of industrial fill material will be submitted for analysis of PAHs, VOCs and/or RCRA metals. The selection of samples to submit for analysis will utilize a biased sampling approach that will rely on a variety of specific site observations	F-7/F-8	Soil samples will be screened utilizing a variety of visual observations and using a PID instrument to identify select samples for additional analysis. PID readings will be used to identify samples for additional analysis.
Groundwater monitoring	Groundwater	PCBs, RCRA Metals, Low-Level Mercury, VOCs, PAHs	Low-Level (Mercury) Normal (All Others)	Twelve groundwater monitoring wells are to be installed onsite. Three of the wells will represent upgradient groundwater quality. A sample will be collected from each well plus 1 field duplicate and trip blank.	F-9	To provide an initial site groundwater monitoring well network, site groundwater flow direction, and determination of downgradient impacts to groundwater.

Footnotes:

(1) See Project Sampling SOP References table (Worksheet #21).

(2) The actual number of soil samples will be determined using a systematic random sampling strategy, following the MDEQ's S^{3}TM guidance (MDEQ, 2002).

QAPP Worksheet #19
Analytical SOP Requirements

MATRIX	ANALYTICAL GROUP	CONCENTRATION LEVEL	ANALYTICAL AND PREPARATION METHOD/SOP REFERENCE[1]	SAMPLE VOLUME	CONTAINERS (number, size, and type)	PRESERVATION REQUIREMENTS (chemical, temperature, light protected)	MAXIMUM HOLDING TIME (preparation/ analysis)
Water	PCBs	All	W-1 (AM E-608)	2 liters	Two 1-liter amber glass bottles with Teflon®-lined lid	Cool to 4°C ±2°	14 days to extraction; 40 days to analysis
Water	TSS	All	W-2, W-5 (AM E-160.2) (AQ-O-DLYCHK)	1 liter	One 1-liter plastic container	Cool to 4°C ±2°	7 days to analysis
Water	Total phosphorus	All	W-3 (AM E-365.3)	500 mL	One 500-mL plastic container	H_2SO_4 to pH<2; cool to 4°C	28 days to analysis
Water	TAL metals (total)	All	W-14 (AM E-200.8M)	500 mL	1 x 1,000-mL high-density polyethylene bottle	Cool to 4°C, add nitric acid to pH <2	6 months
Water	Total cyanide and amenable cyanide	All	W-25 (AM E-335.2 CLP-M)	500 ml	1 x 500 ml plastic bottle	Sodium Hydroxide to pH ≤ 12; Cool to 4°C	14 days
Water	Low-level mercury (total)	All	C-4 (MET-1631)	500 mL	1 x 500-mL fluorinated LPE bottle	Add HCl to pH <2, 2.5 mL of BrCl is added at laboratory	48 hrs unpreserved; 90 days preserved to analysis.
Water	SVOCs	All	W-15 (AM E-8270)	1,000 mL	2 x 1,000-mL amber glass bottle, Teflon®-lined cap	Cool to 4°C; protect from light	Extract within 7 days; analyze within 40 days of extraction
Water	VOCs	All	W-21 (AM E-8260)	1 x 40 mL VOA vial	3 x 40-mL glass VOA vials with Teflon® septum	Cool to 4°C, add HCl to pH <2; protect from light	14 days (sample should remain on-site less than 24 hours)
Soil/Sediment	PCBs	All	W-4 (AM-E-8082)	200 grams	One 8-oz glass jar with Teflon®-lined lid	Cool to 4°C ±2°	14 days to extraction; 40 days to analysis

QAPP Worksheet #19
Analytical SOP Requirements

MATRIX	ANALYTICAL GROUP	CONCENTRATION LEVEL	ANALYTICAL AND PREPARATION METHOD/SOP REFERENCE[1]	SAMPLE VOLUME	CONTAINERS (number, size, and type)	PRESERVATION REQUIREMENTS (chemical, temperature, light protected)	MAXIMUM HOLDING TIME (preparation/ analysis)
Soil	Metals	All	W-14 (AM E-200.8M)	50 grams	One 500-mL amber glass bottle with Teflon®-lined lid	Cool to 4°C ±2°	6 months
Soil	Cyanide	All	W-25 (AM E-335.2 CLP-M)	10 grams (at least)	One 8-oz wide mouth glass jar	Cool to 4°C ±2°	14 days
Soil	SVOCs	All	W-15 (AM E-8270)	100 grams	One 500-mL amber glass bottle with Teflon®-lined lid	Cool to 4°C ±2°	14 days
Soil	VOCs	All	C-5, C-6 (VOC-8260, VOC-5035)	40 mL	3x40-mL VOA vials	Vial containing 5 ml Methanol, cool to 4ºC ±2º; or bulk soil jar, no head space, cool to 4ºC ±2º	14 days
Soil	GRO, DRO	All	W-22, W-23 (AM U-WTPH-D, AM U-WTPH-G)	100 grams	WTPH-D- One 500-mL amber glass bottle with Teflon®-lined lid. WTPH-G-4 oz jar with Teflon®-lined lid	Cool to 4°C ±2°	14 days
Free product	PCBs	All	S-1	3L	3 x 1L Glass	None	14 days to extraction 40 days to extract analysis
Free product	Viscosity	Not Applicable	S-2	1L	1L Plastic	None	None
Free Product	Specific Gravity	Not Applicable	S-3	500 mL	500 mL Plastic	None	None

Footnote:

[1] See the Analytical SOP Reference (Worksheet #23).

QAPP Worksheet #20
Field Quality Control Sample Summary

MATRIX	ANALYTICAL GROUP	CONCENTRATION LEVEL	ANALYTICAL AND PREPARATION SOP REFERENCE[1]	NUMBER OF SAMPLING LOCATIONS	NUMBER OF FIELD DUPLICATE PAIRS	MATRIX SPIKES	NUMBER OF FIELD BLANKS	NUMBER OF EQUIPMENT BLANKS	TOTAL NUMBER OF SAMPLES TO LABORATORY
Water	PCB	All	W-1	TBD	1/10	1/20	TBD	1/10	TBD
Water	TSS	All	W-2, W-5	TBD	1/10	NA	TBD	1/10	TBD
Water	Total phosphorus	All	W-3	TBD	1/10	1/20	TBD	1/10	TBD
Water	TAL metals (total)	All	W-14	TBD	1/10	1/20	TBD	1/10	TBD
Water	Total cyanide and amenable cyanide	All	W-25	TBD	1/10	1/20	TBD	1/10	TBD
Water	Low-level mercury (total)	All	C-4	TBD	1/10	1/20	TBD	1/10	TBD
Water	SVOCs	All	W-15	TBD	1/10	1/20	TBD	1/10	TBD
Water	VOCs	All	W-21	TBD	1/10	1/20	TBD	1/10	TBD
Soil/Sediment	PCB	All	W-4	TBD	1/10	1/20	TBD	1/10	TBD
Soil	Metals	All	W-14	TBD	1/10	1/20	TBD	1/10	TBD
Soil	SVOCs	All	W-15	TBD	1/10	1/20	TBD	1/10	TBD
Soil	VOCs	All	W-21	TBD	1/10	1/20	TBD	1/10	TBD
Soil	GRO, DRO	All	W-22, W-23	TBD	1/10	NA	TBD	1/10	TBD
Free product	PCBs	All	S-1	TBD	1/10	1/20	TBD	1/10	TBD
Free product	Viscosity	Not Applicable	S-2	TBD	NA	NA	NA	NA	TBD
Free Product	Specific Gravity	Not Applicable	S-3	TBD	NA	NA	NA	NA	TBD

Footnote:
[1] See Analytical SOP Reference (Worksheet #23).

Note:
NA = not applicable.

TBD = to be determined

QAPP Worksheet #21
Project Sampling SOP Reference

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	ORIGINATING ORGANIZATION	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK? (Y/N)	COMMENTS
F-1	Water Sampling and Field Measurement Procedures	RMT	Kemmerer water sampler, peristaltic pump, ISCO automated sampler, YSI 6920 sonde unit, appropriate sample containers	Y	Sample method is dependent on water conditions at the sample location and specified analysis required
F-2	Surface Water Flow Measurement Procedures	RMT	electromagnetic velocity meter	Y	--
F-3	Water Treatment System Monitoring Procedures	RMT	appropriate sample containers	Y	--
F-4	Sediment Sampling Procedures	RMT	Trowel, stainless steel scoop, bucket auger, tube auger, coring device	Y	Sample method is dependent of sediment site characteristics
F-5	Soil Sampling Procedures Direct Push Sampler	RMT	--	Y	--
F-6	Surficial Soil Sampling Procedures	RMT	Trowel, stainless steel scoop	--	Confirmation sampling will consist of collecting surficial soil samples
F-7	Soil Gas Monitoring	RMT	Geoprobe® rods utilizing the Post Run Tubing (PRT) system and drive point; tubing; Landtec gas monitor	Y	--
F-8	Groundwater Sampling Procedures	To be determined	--	--	--

QAPP Worksheet #22
Field Equipment Calibration, Maintenance, Testing, and Inspection

FIELD EQUIPMENT	CALIBRATION ACTIVITY	MAINTENANCE ACTIVITY	TESTING ACTIVITY	INSPECTION ACTIVITY	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	RESPONSIBLE PERSON	SOP REFERENCE[1]
Multi-parameter Sonde	F-1	Per SOP and manufacturer's specifications	Turbidity, temperature, pH, conductivity, DO	Check membranes and verify accurate operation	Weekly	F-1	F-1	F-1	YSI Environmental Operations Manual
ISCO sampler	Verify sample volumes	Battery charging	Collecting appropriate sample volumes	Check battery operation and tubing	Daily	F-1	F-1	F-1	F-1
Landtec gas sampler, or equivalent	F-7	Per manufacturer's specifications	Measuring concentration of methane, carbon dioxide, and oxygen	Check tubing and sample port integrity	Daily	F-7	F-7	F-7	F-7
Magnehelic pressure gauge, or equivalent	F-7	Per manufacturer's specifications	Measuring pressures	Check tubing and sample port integrity	Daily	F-7	F-7	F-7	F-7
Photoionization Detector	F-8	Per manufacturer's specifications	Measuring photoionization in soil	Check sample device	Daily	F-8	F-8	F-8	F-8
Flow-Through Cell	F-9	Per manufacturer's specifications	temperature, turbidity, dissolved oxygen, specific electrical conductance, and pH electrodes	Check sample device	Daily	F-9	F-9	F-9	F-9
Peristaltic Pump	F-9	Per manufacturer's specifications		Check sample device	Daily	F-9	F-9	F-9	F-9
Water Level Meter	F-9	Per manufacturer's specifications	Water levels	Check sample device	Daily	F-9	F-9	F-9	F-9

[1] See Project Sampling SOP Reference table (Worksheet #21).

QAPP Worksheet #23
Analytical SOP References

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)	ADDITIONAL EPA COMMENTS (BEYOND EXPIRATION DATE)?
W-1	Organochlorine and PCB (aroclor) pesticide analysis by GC/ECD, AM E-608, September 11, 2007	Definitive	Total PCB in water	Gas chromatography/ Electron capture detectors (GC/ECD)	Weyerhaeuser Analysis and Testing Services (WATS)	N	No – Approved by EPA with last version of Multi-Area QAPP
W-2	Suspended solids in water and wastewater. AM E-160.2, July 13, 2007	Definitive	TSS	Balance	WATS	N	No – Approved by EPA with last version of Multi-Area QAPP
W-3	Total and total soluble phosphorus in water, AM E-365.3, August 19, 2008	Definitive	Total phosphorus as P in water	Spectrophotometer	WATS	N	No – Approved by EPA with last version of Multi-Area QAPP
W-4	Polychlorinated biphenyl analysis by GC/ECD, AM E-8082, December 4, 2007	Definitive	Total PCB in soil and sediment	GC/ECD	WATS	N	No – Approved by EPA with last version of Multi-Area QAPP
W-5	Daily balance, water, and temperature checks, AQ-O-DLYCHK, February 21, 2007	Definitive	TSS	Balance	WATS	N	No – Approved by EPA with last version of Multi-Area QAPP
W-6	General Procedures for Performing Chromatographic Separations and Calibrations, AL G-8000, September 11, 2007	Definitive	General Procedures	Not Applicable	WATS	N	No – Approved by EPA with last version of Multi-Area QAPP
W-7	Sample Management Procedures, OP S-SMO, July 13, 2007	Definitive	Sample Management	Not Applicable	WATS	N	No – Approved by EPA with last version of Multi-Area QAPP
W-8	Procedure for Training Personnel to Perform Test Methods and Standard Operating Procedures. OQ TRAIN. August 19, 2008.	Not applicable	Training	Not Applicable	WATS	N	No – Approved by EPA with last version of Multi-Area QAPP

QAPP Worksheet #23
Analytical SOP References

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)	ADDITIONAL EPA COMMENTS (BEYOND EXPIRATION DATE)?
W-9	Analytical Procedure 3500, AC G-3500, August 15, 2008	Definitive	PCBs and SVOCs in water	Not Applicable	WATS	N	Yes – But revised by WATS 7/07, per comment 10.6 in 6/07 EPA Letter
W-10	Extraction by Continuous Liquid-Liquid Extractor, AC G-3520, August 19, 2008	Definitive	VOCs, SVOCs, and PCBs in water(1)	Not Applicable	WATS	N	Yes – See Footnote (1) per Comment 10.8 in 6/07 EPA Letter.
W-11	Sonication for Soil and Sediments, AC G-3550, July 13, 2007	Definitive	PCBs and SVOCs in soil and sediment(2)	Not Applicable	WATS	N	Yes – See Footnote (2) per comment 10.7 in 6/07 EPA Letter
W-12	Acid Digestion of Aqueous Samples & Extracts for Metals Analysis by ICP & ICPMS, AM E-3010, March 6, 2007	Definitive	Metals in water	Not Applicable	WATS	N	No – Comment 12 in 6/07 EPA letter was suggestion only
W-13	Acid Digestion of Sediments, Soils, Sludges, and Other Solid Samples for Analysis by ICP & ICPMS, AM E-3050, March 6, 2007	Definitive	Metals in soil	Not Applicable	WATS	N	No - Comment 13 in 6/07 EPA letter was suggestion only
W-14	Metals Analysis by ICPMS. AM E-200.8M. August 15, 2008	Definitive	Metals in water and soil	Inductively Coupled Plasma Mass Spectrometer (ICPMS)	WATS	N	Yes – Replaced per Comment 16 in 6/07 EPA Letter
W-15	Capillary GC/MS for Semivolatile Organics. AM E-8270. July 20, 2007	Definitive	SVOCs in water and soil	GC/MS, capillary column	WATS	N	Yes – But revised by WATS 7/07, per comment 10.10 in 6/07 EPA Letter
W-16	Total Solids in Solid and Semi-Solid Samples. AM S-2540G. August 19, 2008.	Definitive	Total solids in soil and sediment	Scale	WATS	N	Added per U.S. EPA 6/07 Comments

QAPP Worksheet #23
Analytical SOP References

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)	ADDITIONAL EPA COMMENTS (BEYOND EXPIRATION DATE)?
W-17	Procedure for Florisil Clean Up of Organic Extracts. AC G-3620. September 11, 2007.	Definitive	PCBs in water, soil, and sediment	Not applicable	WATS	N	Added per U.S. EPA 6/07 Comments
W-18	Gel Permeation Clean-Up. AP E-3640. August 19, 2008.	Definitive	PCBs and SVOCs in water, soil, and sediment	Not applicable	WATS	N	Added per U.S. EPA 6/07 Comments
W-19	Condensation and Evaporation Techniques. AC G-CONC. August 19, 2008.	Definitive	PCBs and SVOCs in water, soil, and sediment	Not applicable	WATS	N	Added per U.S. EPA 6/07 Comments
W-20	Organic Laboratory Analytical Standards. AC G-ORGSTD. July 18, 2007.	Definitive	VOCs, SVOCs, and PCBs in water, soil, and sediment	Not applicable	WATS	N	Added per U.S. EPA 6/07 Comments
W-21	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, AM E-8260. August 27, 2008.	Definitive	VOCs in water	GC/MS	WATS	N	
W-22	Diesel and Motor Oil Range Compounds, Analysis of. AM U-WTPH-D. August 20, 2008.	Definitive	Diesel range compounds	GC/FID, Capillary column	WATS	N	
W-23	Gasoline Range Compounds, Analysis of. AM U-WTPH-G. August 19, 2008.	Definitive	Gasoline range compounds	GC/FID, megabore column	WATS	N	
W-24	Cyanide, Total, AM E-335.2 CLP-M. August 27, 2008	Definitive	Cyanide	Spectrophotometer	WATS	N	
C-1	Sample Disposal, SMO-SDIS, Rev 7, March 26, 2008	Not Applicable	Sample Disposal	Not Applicable	Columbia Analytical Services (CAS)	N	
C-2	Sample Tracking and Internal Chain-of-Custody, SMO-SCOC, Rev 11, March 21, 2008	Not Applicable	Chain-of-Custody	Not Applicable	CAS	N	
C-3	Sample Receiving, SMO-GEN, Rev 24, March 5, 2008	Not Applicable	Sample Receiving	Not Applicable	CAS	N	

QAPP Worksheet #23
Analytical SOP References

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)	ADDITIONAL EPA COMMENTS (BEYOND EXPIRATION DATE)?
C-4	Mercury in Water by Atomic Fluorescence Spectrometry, MET-1631, Rev 9. March 21, 2008	Definitive	Low-flow mercury in water	Cold-Vapor Atomic Fluorescence Spectrometer (CVAFS)	CAS	N	
C-5	Volatile Organic Compounds by GC/MS. VOC-8260, REV 12. March 21, 2008.	Definitive	VOCs in soil or water	GC/MS, Capillary column	CAS	N	
C-6	Purge and Trap/extraction for volatile organics in soil and waste samples, closed system. VOC-5035, REV 7. February 1, 2008.	Definitive	VOCs in soil and waste	Not applicable	CAS	N	
S-1	Method 8082 for Polychlorinated Biphenyls (PCBs) by Gas Chromatography. SOP 8082, REV 4, May 21, 2007	Definitive	PCBs in oil	GC/ECD	Summit Environmental Testing (SET)	N	
S-2	ASTM Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids	Definitive	Free product/oil viscosity	Viscometer	SET	N	
S-3	ASTM Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)	Definitive	Specific Gravity	Hydrometer	SET	N	

Notes:
(1) See appropriate analytical method for a discussion of how the extract will be stored if not immediately analyzed.
(2) Volumes of surrogates and matrix spiking are logged in the laboratory standard log book.

QAPP Worksheet #24
Analytical Instrument Calibration

INSTRUMENT	CALIBRATION PROCEDURE	FREQUENCY OF CALIBRATION	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON RESPONSIBLE FOR CA	SOP REFERENCE[1]
Cold-Vapor Atomic Fluorescence Spectrometer (CVAFS)	See C-4	At the beginning of each analysis sequence.	See C-4	Correct the problem and repeat the calibration.	Analyst	C-4
ICPMS	See W-14	At the beginning of each analysis sequence. CCV analyzed after calibration, after every 10 samples (max) and at end. Initial calibration blank/verification follow first CCV.	See W-14	Inspect the instrument, correct problems, repeat calibration.	Analyst	W-14
GC/MS	Tuning. See C-5, W-15 & W-21	Before 12 hr. sample analysis.	See C-5, W-15 & W-21	Inspect the instrument, correct problems, re-tune and/or recalibrate	Analyst	See C-5, W-15 & W-21
	Calibration, See C-5, W-15 & W-21	Initial calibration at instrument set-up and when calibration verification or tuning fail criteria. Calibration verification before 12 hr. sample analysis	See C-5, W-15 & W-21	Inspect the instrument, correct problems and recalibrate.	Analyst	See C-5, W-15 & W-21
GC/FID	See W-22 & W-23	Initial calibration at instrument set-up and when calibration verification fails criteria. Check mid-point standards after every 10 samples and at the beginning and end of the analysis.	Initial calibration RSD ≤ 20% For continuing calibration standard must not exceed a percent difference of ± 15	Inspect system, correct problem, and rerun calibration on affected samples.	Analyst	See W-22 & W-23

QAPP Worksheet #24
Analytical Instrument Calibration

INSTRUMENT	CALIBRATION PROCEDURE	FREQUENCY OF CALIBRATION	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON RESPONSIBLE FOR CA	SOP REFERENCE[1]
GC/ECD	See W-1, W-4 & S-1	After initial calibration, a check standard of the match-point Aroclor 1016/1260 mixture is analyzed after every 10 samples and at the end of the sequence.	Initial calibration RSD for 1016/1260 ≤ 20% or linear regression >0.99 Continuing calibration standard must not exceed a percent difference of ± 15	Inspect system, correct problem, and rerun calibration on affected samples.	Analyst	See W-1, W-4 & S-1
Balance	See SOP W-5.	Daily	NA	Inspect system, correct problem, and rerun calibration on affected samples.	Analyst	W-2 and W-5
Spectrophotometer	Construct two 6-point calibration curves of absorbance versus concentration for low and high levels.	Before sample analysis, when check standard is outside Confidence Limits; also, after major instrument maintenance	Initial calibration correlation coefficient >0.995 ICV/CCV 90-110%	Fix problem and recalibrate.	Analyst	W-3, W-24
Hydrometer	Known Standard	Weekly	± 10% of Standard	See S-2	Analyst	S-2
Viscometer	Known Standard	Weekly	± 10% of Standard	See S-3	Analyst	S-3

Footnote:
(1) See Analytical SOP References (Worksheet #23).

QAPP Worksheet #25
Analytical Instrument and Equipment Maintenance, Testing, and Inspection

INSTRUMENT/ EQUIPMENT	MAINTENANCE ACTIVITY	TESTING ACTIVITY	INSPECTION ACTIVITY	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	RESPONSIBLE PERSON	SOP REFERENCE[1]
GC/ECD	▪ Change septa weekly, or as needed. ▪ Change gas line dryers as needed. ▪ Replace injection port liner weekly, or as needed. ▪ Clip column. ▪ Replace GC column as needed. ▪ Clean/Replace detector as needed. ▪ Provide that gas supply is sufficient and delivery pressure is adequate.	Conduct leak test.	Check connections and bake out instrument.	W-1 and W-4	W-1 and W-4	Inspect system, correct problem, rerun calibration, and reanalyze affected samples.	Analyst	W-1 and W-4
Spectro-photometer	▪ Dust lamp and front of front lens. ▪ Clean sample compartment. ▪ Clean windows. ▪ Clean cuvettes.		Check the zero.	Daily	See W-3, W-24.	Inspect system, correct problem, and rerun calibration on affected samples.	Analyst	W-3, W-24

QAPP Worksheet #25
Analytical Instrument and Equipment Maintenance, Testing, and Inspection

INSTRUMENT/ EQUIPMENT	MAINTENANCE ACTIVITY	TESTING ACTIVITY	INSPECTION ACTIVITY	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	RESPONSIBLE PERSON	SOP REFERENCE[1]
ICPMS	See ICPMS Instruction Manual		Check gas cylinder pressures, recirculator is 20ºC, allow 30-min warm-up after ignition, run performance report, tune instrument.	W-14	W-14	Inspect system, resolve any deficiencies, rerun calibration on affected samples	Analyst	W-14
Balance	▪ Professional service contract		NA	Once/year	NA	NA	Service contractor	NA
Cold-Vapor Atomic Fluorescence Spectrometer (CVAFS)	See C-4		See C-4	See C-4	See C-4	Inspect system, correct problems, re-run calibration and affected samples if necessary	Analyst	C-4
Hydrometer	Clean hydrometer and cylinder with mineral spirits after each use		Check for damaged hydrometer	S-2	See S-2	Reanalyze sample	Analyst	S-2
Viscometer	Clean viscosity tube with mineral spirits after each use.		Check for damage or capillary blockage	S-3	See S-3	Reanalyze sample	Analyst	S-3

Footnote:

[1] See Analytical SOP References (Worksheet #23).

QAPP Worksheet #26
Sample Handling System (WATS)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (personnel/organization): Jennifer Overvoorde, RMT
Sample Packaging (personnel/organization): Jennifer Overvoorde, RMT
Coordination of Shipment (personnel/organization): Jennifer Overvoorde, RMT
Type of Shipment/Carrier: Overnight courier
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (personnel/organization): Dennis Catalano, WATS (or designee)
Sample Custody and Storage (personnel/organization): Dennis Catalano, WATS (or designee)
Sample Preparation (personnel/organization): Dennis Catalano, WATS (or designee)
Sample Determinative Analysis (personnel/organization): Dennis Catalano, WATS (or designee)
SAMPLE ARCHIVING
Field Sample Storage (number of days from sample collection): 30 days from submittal of final report
Sample Extract/Digestate Storage (number of days from extraction/digestion): 60 days from submittal of final report
Biological Sample Storage (number of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Dennis Catalano, WATS
Number of Days from Analysis: 60 days minimum from submittal of final report

QAPP Worksheet #27
Sample Custody Requirements

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

- The field sampler is personally responsible for the care and custody of the samples until they are transferred to the laboratory or properly dispatched. Keep the number of people handling the samples to a minimum to ensure proper field Chain-of-Custody.
- Field Chain-of-Custody Records will accompany all analytical samples and sample shipping containers to document their transfer from the field to the analytical laboratory. The procedures to be implemented are as follows:
  - Complete Chain-of-Custody Records Indicating sample identification, containers filled, sampling date, sampling time, sample collector's name, and sample preservation, if applicable. Also note this information in the field notebooks.
  - Repack shipping containers with samples, Chain-of-Custody Records, and water ice. Assign a Chain-of-Custody Record to each set of sample containers to be shipped.
  - Place completed Chain-of-Custody Records in a plastic bag, seal the bag, and tape it to the inside cover of the shipping container. After the samples are iced, add the date to the Chain-of-Custody Record, seal the coolers with strapping tape, add custody seals, and ship the coolers to WATS Laboratory using an overnight delivery service. Identify common carriers or intermediate individuals on the Chain-of-Custody Record, and retain copies of all bills-of-lading. When the samples are received in the laboratory, handle and process them in accordance with laboratory SOPs, or specified analytical methods, as defined in this QAPP.
  - The laboratory receiving the samples will check shipping containers for completeness of paperwork, broken custody seals, damaged sample containers, and sample preservation as specified by the analytical method. The laboratory's sample management staff will note any problems, log the samples into the laboratory, and complete the Chain-of-Custody Record. The person relinquishing the samples to the facility or agency will request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is to be noted in the "Received By" space on the Record.
  - Include copies of the Chain-of-Custody Record with the analytical data.
- A separate sample receipt is prepared whenever samples are split with a government agency. The receipt is marked to indicate with whom the samples are being split. The person relinquishing the samples to the agency should request the agency representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is to be noted on the receipt and in the field notebook.
- A copy of the Chain-of-Custody Record will accompany the samples to the laboratory. The field sampling personnel will retain one copy with the field notes. If a Chain-of-Custody Record Is damaged in shipment, the field copy will be made available. A written statement will be prepared by the person who collected the samples, listing the samples that were recorded on the damaged record, and describing when and how the samples were collected. The statement should include information such as field notebook entries regarding the sample. This statement is submitted to the On-Site Coordinator and the RMT Project Manager for further action, as necessary.

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

The laboratory assigns a unique, sequentially numbered sample code to each sample received. Laboratory custody procedures for sample receiving and log-in, storage, tracking, and holding time requirements are described in the laboratory's Quality Assurance Manual and in the Laboratory SOPs (see Attachment 1-5).

QAPP Worksheet #27 (*continued*)
Sample Custody Requirements

Sample Identification Procedures:

- Label each bottle with the project number, the sample identifier, the sample type, the sampler's initials, and the date and time of sample collection.
- Complete sample labels for each sample and custody seals for each shipment container using waterproof ink, unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because the ink pen would not function in freezing weather.

Chain-of-Custody Procedures:

An example Chain-of-Custody Record from WATS Laboratory is shown in Attachment 1-3. The Records should be legibly completed. Errors will be corrected by drawing a single line through the incorrect information and entering the correct information. All corrections are to be initialed and dated by the person making the correction. This procedure applies to words or figures inserted or added to a previously recorded statement.

The following information must be included on the Chain-of-Custody Record Attachment 1-3:

- Facility name and address, project number, and sampler identification.
- "The Sample ID No. and Description" portion of the Record must be completed for each sample. This information includes the Field Sample ID, sample date and time, and sample depth. The sampling time **MUST** also be noted on the sample bottle (except for blind field duplicates, where date and time would not be noted on the bottle label or Chain-of-Custody Record).
- The sample container type and number, sample matrix, preservative/filtration, and requested analysis must be designated by checking the appropriate box and/or writing the required information.

Sample custody is documented on the lower portion of the Record, and includes the sampler's signature, signatures of persons involved in the possession of the sample with dates and times, and the date on which the sample was received at the laboratory, as described further below.

- ***Relinquished by/Received by*** - This part of the Chain-of-Custody Record is a record of the individuals who actually had the samples in their custody. The spaces must be used in chronological order as the Chain-of-Custody Record is transferred with the samples.
  - (1) Sampler signs when relinquishing custody.
  - (1) Person accepting custody of samples from sampler signs.
  - (2) Person in (1) must sign when relinquishing custody.
  - (2)-(3) These are completed as necessary in the same manner as above.
- ***Sampler*** - The person/persons collecting the samples must sign their name and print their name under their signature, and record the date and time they relinquish the samples to either the laboratory or the shipper. The final signature is that of the person receiving the samples at the laboratory.
- ***Special Instructions*** – The sampler may provide additional information about a sample, *e.g.*, if an odor is present, high or low pH, etc.
- ***Possible Hazard Identification –*** The sampler may include any known or suspected hazards associated with the samples. Sample entry personnel may add information to this section based on communications from the laboratory Project Manager or Supervisor after samples are received. Laboratory Team Leaders will use any hazard information to update and advise their analysts before work is started.

Note: If commercial carriers are used, the name of the carrier, any airbill number, and the date and time of relinquishing the sample containers are written on the airbill by sample entry or field personnel, and the airbill is attached to the Chain-of-Custody Record.

A copy of the Chain-of-Custody Record should be returned with the sample results. The laboratory service request number should be written on the Chain-of-Custody Record to facilitate its use during project data entry.

QAPP Worksheet #28-1
QC Samples - PCBs (Surface Water)

Matrix: Water | Sampling SOP: F-1, F-3 | Field Sampling Organization: RMT

Analytical Group: PCBs | Analytical Method/SOP Reference: W-1 | Analytical Organization: WATS

Concentration Level: All | Sampler's Name: NA | Number of Sample Locations: To be determined[1]

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	1 per 10 samples	RPD <20	Qualify data as needed.	RMT Data Validator	Precision	RPD
Surrogates	2 per sample	Laboratory control limits	Re-extraction/ Reanalysis	Laboratory staff	Accuracy	% Recovery
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-extraction/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per 10 samples	< Laboratory reporting limit	Qualify data as needed.	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-extraction/ Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed.	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

Footnote:

[1] See project-specific scope and Field Sampling Plan.

QAPP Worksheet #28-2
QC Samples - TSS (Surface Water)

Matrix: Water | Sampling SOP: F-3 | Field Sampling Organization: RMT

Analytical Group: TSS | Analytical Method/SOP Reference: W-2, W5 | Analytical Organization: WATS

Concentration Level: All | Sampler's Name: NA | Number of Sample Locations: To be determined(1)

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	1 per 10 samples	RPD <20	Qualify data as needed.	RMT Data Validator	Precision	RPD
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-extraction/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per 10 samples	< Laboratory reporting limit	Qualify data as needed.	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-extraction/ Reanalysis	Laboratory staff	Accuracy	% Recovery

Footnote:
(1) See project-specific scope and Field Sampling Plan.

QAPP Worksheet #28-3
QC Samples - Phosphorus (Surface Water)

Matrix: Water | Sampling SOP: F-3 | Field Sampling Organization: RMT

Analytical Group: Total phosphorus | Analytical Method/SOP Reference: W-3 | Analytical Organization: WATS

Concentration Level: All | Sampler's Name: NA | Number of Sample Locations: To be determined[1]

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	1 per 10 samples	RPD <20	Qualify data as needed.	RMT Data Validator	Precision	RPD
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-extraction/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per 10 samples	< Laboratory reporting limit	Qualify data as needed.	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-extraction/ Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed.	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

Footnote:

[1] See project-specific scope and Field Sampling Plan.

QAPP Worksheet #28-4
QC Samples - PCBs (Soil/Sediment)

Matrix: Soil/Sediment | Sampling SOP: F-4 | Field Sampling Organization: RMT

Analytical Group: PCBs | Analytical Method/SOP Reference: W-4 | Analytical Organization: WATS

Concentration Level: All | Sampler's Name: NA | Number of Sample Locations: To be determined[(1)]

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	1 per 10 samples	RPD <50	Qualify data as needed.	RMT Data Validator	Precision	RPD
Surrogates	2 per sample	Laboratory control limits	Re-extraction/ Reanalysis	Laboratory staff	Accuracy	% Recovery
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-extraction/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per 10 samples	< Laboratory reporting limit	Qualify data as needed.	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-extraction/ Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed.	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

Footnote:

(1) See project-specific scope and Field Sampling Plan.

QAPP Worksheet #28-5
QC Samples - PCBs (Free Product)

Matrix: Free Product | Sampling SOP: F-1, F-3 | Field Sampling Organization: RMT

Analytical Group: PCBs | Analytical Method/SOP Reference: S-1 | Analytical Organization: SET

Concentration Level: All | Sampler's Name: NA | Number of Sample Locations: To be determined[1]

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	1 per 10 samples	RPD < 100%	Qualify data as needed.	RMT Data Validator	Precision	RPD
Surrogates	1 per sample	Laboratory control limits	Re-extraction/ Reanalysis	Laboratory staff	Accuracy	% Recovery
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-extraction/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per 10 samples	< Laboratory reporting limit	Qualify data as needed.	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-extraction/ Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed.	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

Footnote:

[1] See project-specific scope and Field Sampling Plan.

QAPP Worksheet #28-6
QC Samples – TAL Metals (Groundwater)

Matrix: Water | Sampling SOP: F-8 | Field Sampling Organization: RMT

Analytical Group: TAL Metals | Analytical Method/SOP Reference: W-14 | Analytical Organization: WATS

Concentration Level: All (Except Low-Level Mercury) | Sampler's Name: NA | Number of Sample Locations: See project-specific scope and Field Sampling Plan

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	1 per ten samples	RPD < 100%	Qualify data as needed	RMT Data Validator	Precision	RPD
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-digestion/ reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per ten samples	< Laboratory reporting limit	Qualify data as needed	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-digestion/ reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

QAPP Worksheet #28-7
QC Samples – TAL Metals (Soil)

Matrix: Soil | Sampling SOP: F-5, F-6 | Field Sampling Organization: RMT

Analytical Group: TAL Metals and Cyanide | Analytical Method/SOP Reference: W-14 | Analytical Organization: WATS

Concentration Level: All | Sampler's Name: NA | Number of Sample Locations: See project-specific scope and Field Sampling Plan

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	1 per ten samples	RPD < 100%	Qualify data as needed	RMT Data Validator	Precision	RPD
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-digestion/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per ten samples	< Laboratory reporting limit	Qualify data as needed	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-digestion/ Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

QAPP Worksheet #28-8
QC Samples – Cyanide (Groundwater)

Matrix: Water | Sampling SOP: F-8 | Field Sampling Organization: RMT

Analytical Group: Cyanide | Analytical Method/SOP Reference: W-24 | Analytical Organization: WATS

Concentration Level: Total | Sampler's Name: NA | Number of Sample Locations: See project-specific scope and Field Sampling Plan

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	1 per ten samples	RPD < 100%	Qualify data as needed	RMT Data Validator	Precision	RPD
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-digestion/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per ten samples	< Laboratory reporting limit	Qualify data as needed	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-digestion/ Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

QAPP Worksheet #28-9
QC Samples – Cyanide (Soil)

Matrix: Soil | Sampling SOP: F-8 | Field Sampling Organization: RMT

Analytical Group: Cyanide | Analytical Method/SOP Reference: W-24 | Analytical Organization: WATS

Concentration Level: Total | Sampler's Name: NA | Number of Sample Locations: See project-specific scope and Field Sampling Plan

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	1 per ten samples	RPD < 100%	Qualify data as needed	RMT Data Validator	Precision	RPD
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-digestion/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per ten samples	< Laboratory reporting limit	Qualify data as needed	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-digestion/ Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

QAPP Worksheet #28-10
QC Samples – Low Level Mercury (Groundwater)

Matrix: Water | Sampling SOP: F-8 | Field Sampling Organization: RMT

Analytical Group: Mercury | Analytical Method/SOP Reference: C-4 | Analytical Organization: CAS

Concentration Level: Low-Level | Sampler's Name: NA | Number of Sample Locations: See project-specific scope and Field Sampling Plan

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	1 per ten samples	RPD < 100%	Qualify data as needed	RMT Data Validator	Precision	RPD
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-digestion/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per ten samples	< Laboratory reporting limit	Qualify data as needed	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-digestion/ Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

QAPP Worksheet #28-11
QC Samples - SVOCs (Groundwater)

Matrix: Water | Sampling SOP: F-8 | Field Sampling Organization: RMT

Analytical Group: SVOCs | Analytical Method/SOP Reference: W-15 | Analytical Organization: WATS

Concentration Level: All | Sampler's Name: NA | Number of Sample Locations: See project-specific scope and Field Sampling Plan

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	1 per ten samples	RPD < 100%	Qualify data as needed	RMT Data Validator	Precision	RPD
Surrogates	6 per sample	Laboratory control limits	Re-extraction/ Re-analysis	Laboratory Staff	Accuracy	% Recovery
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-extraction/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per ten samples	< Laboratory reporting limit	Qualify data as needed	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-extraction/ Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

QAPP Worksheet #28-12
QC Samples - SVOCs (Soil)

Matrix: Soil | Sampling SOP: F-5, F-6 | Field Sampling Organization: RMT

Analytical Group: SVOCs | Analytical Method/SOP Reference: W-15 | Analytical Organization: WATS

Concentration Level: All | Sampler's Name: NA | Number of Sample Locations: See project-specific scope and Field Sampling Plan

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	1 per ten samples	RPD < 100%	Qualify data as needed	RMT Data Validator	Precision	RPD
Surrogates	6 per sample	Laboratory control limits	Re-extraction/ Re-analysis	Laboratory Staff	Accuracy	% Recovery
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-extraction/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per ten samples	< Laboratory reporting limit	Qualify data as needed	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Re-extraction/ Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

QAPP Worksheet #28-13
QC Samples - VOCs (Groundwater)

Matrix: Water | Sampling SOP: F-8 | Field Sampling Organization: RMT

Analytical Group: VOCs | Analytical Method/SOP Reference: W-21 | Analytical Organization: WATS

Concentration Level: All | Sampler's Name: NA | Number of Sample Locations: See project-specific scope and Field Sampling Plan

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	1 per ten samples	RPD < 100%	Qualify data as needed	RMT Data Validator	Precision	RPD
Surrogates	3 per sample	Laboratory control limits	Re-analysis	Laboratory Staff	Accuracy	% Recovery
Method blanks	1 per analytical batch	< Laboratory reporting limit	Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per ten samples	< Laboratory reporting limit	Qualify data as needed	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

QAPP Worksheet #28-14
QC Samples - VOCs (Soil)

Matrix: Soil | Sampling SOP: F-5, F-6 | Field Sampling Organization: RMT

Analytical Group: VOCs | Analytical Method/SOP Reference: C-5 | Analytical Organization: CAS

Concentration Level: All | Sampler's Name: NA | Number of Sample Locations: See project-specific scope and Field Sampling Plan

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	1 per ten samples	RPD < 100%	Qualify data as needed	RMT Data Validator	Precision	RPD
Surrogates	4 per sample	Laboratory control limits	Re-analysis	Laboratory Staff	Accuracy	% Recovery
Method blanks	1 per analytical batch	< Laboratory reporting limit	Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per ten samples	< Laboratory reporting limit	Qualify data as needed	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

QAPP Worksheet #28-15
QC Samples – DRO & GRO (Soil)

Matrix: Soil | Sampling SOP: F-5, F-6 | Field Sampling Organization: RMT

Analytical Group: DRO & GRO | Analytical Method/SOP Reference: W-22& W-23 | Analytical Organization: WATS

Concentration Level: All | Sampler's Name: NA | Number of Sample Locations: See project-specific scope and Field Sampling Plan

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	1 per ten samples	RPD < 100%	Qualify data as needed	RMT Data Validator	Precision	RPD
Surrogates	1 per sample (DRO) 2 per sample (GRO)	Laboratory control limits	Re-extraction/ Reanalysis	Laboratory Staff	Accuracy	% Recovery
Method blanks	1 per analytical batch	< Laboratory reporting limit	Re-extraction/ Reanalysis	Laboratory staff	Bias/Contamination	< Laboratory reporting limit
Equipment blanks	1 per ten samples	< Laboratory reporting limit	Qualify data as needed	RMT Data Validator	Bias/Contamination	< Laboratory reporting limit
Laboratory control sample (LCS)	1 per analytical batch	Laboratory control limits	Reanalysis	Laboratory staff	Accuracy	% Recovery
MS/MSD	1 per 20 samples	Laboratory control limits	Qualify data as needed	Laboratory staff RMT Data Validator	Accuracy/Precision	% Recovery/RPD

QAPP Worksheet #29
Project Documents and Records

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 ▪ Sampling logs ▪ Chain-of-Custody Records ▪ Air bills ▪ Custody seals	▪ Equipment calibration logs ▪ Field data records ▪ Field instrument maintenance logs	▪ Sample receipt, custody, and tracking records ▪ Standard traceability logs ▪ Equipment calibration logs ▪ Sample prep logs ▪ Run logs ▪ Equipment maintenance, testing, and inspection logs ▪ Corrective action forms ▪ Reported field sample results ▪ Reported results for standards, QC checks, and QC samples ▪ Instrument printouts (raw data) for field sample standards, QC checks, and QC sample ▪ Data package completeness checklists ▪ Sample disposal records ▪ Extraction/Cleanup records ▪ Raw data (stored on diskette or CD-R) ▪ Analytical reports	▪ Data validation checklists ▪ Data quality assessments	▪ Consent Decree documents ▪ Progress reports to the U.S. EPA ▪ Work plans and Field Sampling Plans ▪ Health and Safety Plans ▪ Quality Assurance Project Plan ▪ Quality Management Plan ▪ Remedial Investigation and Risk Assessment Reports ▪ Feasibility Studies ▪ Design Reports ▪ Construction Documentation Reports ▪ Monitoring Reports

QAPP Worksheet #30
Analytical Services

MATRIX	ANALYTICAL GROUP	CONCENTRATION LEVEL	SAMPLE LOCATIONS/ ID NUMBERS	ANALYTICAL SOP	DATA PACKAGE TURNAROUND TIME	LABORATORY/ ORGANIZATION (name, address, contact person, and telephone number)	BACKUP LABORATORY/ ORGANIZATION (name, address, contact person, and telephone number)
Ground/ Surface water	PCBs, TSS, Phosphorus, TAL metals, Total Cyanide, SVOCs, VOCs,	All	TBD	W-1, W-2, W-3, W-5, W-14, W-15, W-21, W-24	Standard turnaround time (17 calendar days)	Weyerhaeuser Analysis and Testing Services (WATS) Weyerhaeuser Company Mail Stop: WTC2F25 32901 Weyerhaeuser Way Federal Way, WA 98001 Dennis Catalano 253.924.6242	Pace Analytical Services, Inc., Wisconsin 1700 Elm Street, SE Suite 200 Minneapolis, MN 55414 Tod Noltenmeyer (608) 232-3300
	Mercury (low level)	All	TBD	C-4	Standard turnaround time (15 calendar days)	Columbia Analytical Services (CAS), Inc 1317 South 13th Avenue Kelso, WA 98626 Jeff Christian 360.557.7222	Pace Analytical Services, Inc., Wisconsin 1700 Elm Street, SE Suite 200 Minneapolis, MN 55414 Tod Noltenmeyer (608) 232-3300
Free Product	Specific Gravity, Viscosity, PCBs	All	TBD	S-1, S-2, S-3	Standard turnaround time (2 calendar days)	Summit Environmental Technologies, Inc (SET) Analytical Laboratories 3310 Win Street Cuyhoga Falls, OH 44223 Ron Gibas 800.278.0140	Pace Analytical Services, Inc., Wisconsin 1700 Elm Street, SE Suite 200 Minneapolis, MN 55414 Tod Noltenmeyer (608) 232-3300

QAPP Worksheet #30
Analytical Services

MATRIX	ANALYTICAL GROUP	CONCENTRATION LEVEL	SAMPLE LOCATIONS/ ID NUMBERS	ANALYTICAL SOP	DATA PACKAGE TURNAROUND TIME	LABORATORY/ ORGANIZATION (name, address, contact person, and telephone number)	BACKUP LABORATORY/ ORGANIZATION (name, address, contact person, and telephone number)
Soil/ Sediment	PCBs, Metals, SVOCs, DRO/GRO	All	TBD	W-4, W-14, W-15, W-22/W-23 W-24	Rush turnaround time (7 calendar days)	Weyerhaeuser Analysis and Testing Services (WATS) Weyerhaeuser Company Mail Stop: WTC2F25 32901 Weyerhaeuser Way Federal Way, WA 9800 Dennis Catalano 253.924.6242	Pace Analytical Services, Inc., Wisconsin 1700 Elm Street, SE Suite 200 Minneapolis, MN 55414 Tod Noltenmeyer (608) 232-3300
	VOCs	All	TBD	C-5, C-6	Standard turnaround time (5 calendar days)	Columbia Analytical Services (CAS), Inc 1317 South 13th Avenue Kelso, WA 98626 Jeff Christian 360.557.7222	Pace Analytical Services, Inc., Wisconsin 1700 Elm Street, SE Suite 200 Minneapolis, MN 55414 Tod Noltenmeyer (608) 232-3300
Third-Party Data Validation	All	All	--	All	--	Environmental Chemistry Consulting Services, Inc. (ECCS) 2525 Advance Road Madison, WI 53718 Greg Graf 608.221.8700	--

Notes:
NA = not applicable.
TBD = to be determined.

QAPP Worksheet #31-1
Planned Project Assessments
(Emergency Response Activities)

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)
Field performance audit	Periodic, based on field schedule	Internal	RMT	Kathy Huibregtse, RMT (Senior Consultant, QA Manager)	Jim Hutchens, RMT (Project Coordinator)	Jennifer Overvoorde, RMT (Field Sampling Coordinator), and Jeff Macri (Construction Manager)	Jim Hutchens, RMT (Project Coordinator)
Field systems audit	Periodic, based on field schedule	Internal	RMT	Tom Stolzenburg, RMT (Data QA Manager)	Jim Hutchens, RMT (Project Coordinator)	Jennifer Overvoorde, RMT (Field Sampling Coordinator), and Jeff Macri (Construction Manager)	Jim Hutchens, RMT (Project Coordinator)
Laboratory audit	As needed, based on laboratory performance	External	RMT	Tom Stolzenburg, RMT (Data QA Manager)	Dennis Catalano, WATS (Laboratory QA/QC Manager)	Dennis Catalano, WATS (Laboratory QA/QC Manager)	Tom Stolzenburg, RMT (Data QA Manager)
Laboratory audit	Per laboratory QA Plan	Internal	WATS	Dennis Catalano, WATS (Laboratory QA/QC Manager)	Dennis Catalano, WATS (Laboratory QA/QC Manager)	Dennis Catalano, WATS (Laboratory QA/QC Manager)	Tom Stolzenburg, RMT (Data QA Manager)

QAPP Worksheet #31-2
Planned Project Assessments
(Predesign Investigations For the Remedial Design For the 12Th Street Landfill)

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)
Field performance audit	Periodic, based on field schedule	Internal	RMT	Kathy Huibregtse, RMT (Senior Consultant, QA Manager)	Linda Hicken, RMT (Project Coordinator)	Jennifer Overvoorde, RMT (Field Sampling Coordinator)	Linda Hicken, RMT (Project Coordinator)
Field systems audit	Periodic, based on field schedule	Internal	RMT	Tom Stolzenburg, RMT (Data QA Manager)	Linda Hicken, RMT (Project Coordinator)	Jennifer Overvoorde, RMT (Field Sampling Coordinator)	Linda Hicken, RMT (Project Coordinator)

QAPP Worksheet #31-3
Planned Project Assessments
(Phase 2 of Addendum No. 1 – Test Pits and Plainwell Mill RI/FS – Phase I Initial Groundwater Assessment)

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)
Field performance audit	Periodic, based on field schedule	Internal	RMT	Kathy Huibregtse, RMT (Senior Consultant, QA Manager)	Jim Hutchens, RMT (Project Coordinator)	Jennifer Overvoorde, RMT (Field Sampling Coordinator), and Jeff Macri (Construction Manager)	Jim Hutchens, RMT (Project Coordinator)
Field systems audit	Periodic, based on field schedule	Internal	RMT	Tom Stolzenburg, RMT (Data QA Manager)	Jim Hutchens, RMT (Project Coordinator)	Jennifer Overvoorde, RMT (Field Sampling Coordinator), and Jeff Macri (Construction Manager)	Jim Hutchens, RMT (Project Coordinator)
Laboratory audit	As needed, based on laboratory performance	External	RMT	Tom Stolzenburg, RMT (Data QA Manager)	Dennis Catalano, WATS (Laboratory QA/QC Manager)	Dennis Catalano, WATS (Laboratory QA/QC Manager)	Tom Stolzenburg, RMT (Data QA Manager)
Laboratory audit	Per laboratory QA Plan	Internal	WATS	Dennis Catalano, WATS (Laboratory QA/QC Manager)	Dennis Catalano, WATS (Laboratory QA/QC Manager)	Dennis Catalano, WATS (Laboratory QA/QC Manager)	Tom Stolzenburg, RMT (Data QA Manager)

QAPP Worksheet #32-1
Assessment Findings and Corrective Action Responses
(Emergency Response Activities)

ASSESSMENT TYPE	NATURE OF DEFICIENCIES DOCUMENTATION	INDIVIDUAL(S) NOTIFIED OF FINDINGS (name, title, organization)	TIME FRAME OF NOTIFICATION	NATURE OF CORRECTIVE ACTION RESPONSE DOCUMENTATION	INDIVIDUAL(S) RECEIVING CORRECTIVE ACTION RESPONSE (name, title, organization)	TIME FRAME FOR RESPONSE
Field performance audit	Checklist	Kathy Huibregtse, RMT (Senior Consultant QA Manager), will notify Jim Hutchens, RMT (Project Coordinator).	Within 72 hours after audit (or sooner, as appropriate)	E-mail response	Jennifer Overvoorde, RMT (Field Sampling Coordinator), and Jeff Macri (Construction Manager)	Within 48 hours after notification (or sooner, as appropriate)
Field systems audit	Checklist	Tom Stolzenburg, RMT (Data QA Manager), will notify Jim Hutchens, RMT (Project Coordinator).	Within 48 hours after audit (or sooner, as appropriate)	E-mail response	Jennifer Overvoorde, RMT (Field Sampling Coordinator), and Jeff Macri (Construction Manager)	Within 48 hours after notification (or sooner, as appropriate)
Internal laboratory audit	Executive Summary from Management Report	Dennis Catalano, WATS (Laboratory QA/QC Manager), will notify Tom Stolzenburg, RMT (Data QA Manager), and appropriate laboratory staff.	Within 48 hours after audit (or sooner, as appropriate)	Executive Summary from Management Report	Greg Graf, RMT (Data QA Manager), and appropriate laboratory staff	Within 48 hours after notification (or sooner, as appropriate)
External laboratory audit	Checklist	Tom Stolzenburg, RMT (Data QA Manager), will notify Dennis Catalano, WATS (Laboratory QA/QC Manager), and Jim Hutchens (Project Coordinator).	Within 1 week after audit	Memorandum	Dennis Catalano, WATS (Laboratory QA/QC Manager)	Within 48 hours after notification (or sooner, as appropriate)

QAPP Worksheet #32-2
Assessment Findings and Corrective Action Responses
(Predesign Investigations For The Remedial Design For the 12th Street Landfill)

ASSESSMENT TYPE	NATURE OF DEFICIENCIES DOCUMENTATION	INDIVIDUAL(S) NOTIFIED OF FINDINGS (name, title, organization)	TIME FRAME OF NOTIFICATION	NATURE OF CORRECTIVE ACTION RESPONSE DOCUMENTATION	INDIVIDUAL(S) RECEIVING CORRECTIVE ACTION RESPONSE (name, title, organization)	TIME FRAME FOR RESPONSE
Field performance audit	Checklist	Kathy Huibregtse, RMT (Senior Consultant QA Manager), will notify Linda Hicken, RMT (Project Coordinator).	Within 72 hours after audit (or sooner, as appropriate)	E-mail response	Jennifer Overvoorde, RMT (Field Sampling Coordinator),	Within 48 hours after notification (or sooner, as appropriate)
Field systems audit	Checklist	Tom Stolzenburg, RMT (Data QA Manager), will notify Linda Hicken, RMT (Project Coordinator).	Within 48 hours after audit (or sooner, as appropriate)	E-mail response	Jennifer Overvoorde, RMT (Field Sampling Coordinator)	Within 48 hours after notification (or sooner, as appropriate)

QAPP Worksheet #33
QA Management Reports

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)
Field audit reports	As needed	As generated	Kathy Huibregtse, Senior Consultant and QA Manager, and Tom Stolzenburg, Data QA Manager	Jennifer Hale, Weyerhaeuser Company Jim Hutchens, RMT, Linda Hicken, RMT
WATS audit (external)	As needed	As generated	Tom Stolzenburg, Data QA Manager	Jennifer Hale, Weyerhaeuser Company Jim Hutchens, RMT, Linda Hicken, RMT
Columbia Analytical Services audit (external)	As needed	As generated	Tom Stolzenburg, Data QA Manager	Jennifer Hale, Weyerhaeuser Company Jim Hutchens, RMT, Linda Hicken, RMT
Summit Environmental Technologies Inc. Analytical Testing audit (external)	As needed	As generated	Tom Stolzenburg, Data QA Manager	Jennifer Hale, Weyerhaeuser Company Jim Hutchens, RMT, Linda Hicken, RMT
Data validation reports	As specified in data assessment section	As generated	Greg Graf, ECCS (third-party data validator)	Jennifer Hale, Weyerhaeuser Company Jim Hutchens, RMT, Linda Hicken, RMT
Data quality summary	As appropriate for data use	As generated	Greg Graf, ECCS (third-party data validator)	Jennifer Hale, Weyerhaeuser Company Jim Hutchens, RMT, Linda Hicken, RMT

QAPP Worksheet #34
Verification (Step I) Process

VERIFICATION INPUT	DESCRIPTION	INTERNAL/ EXTERNAL	RESPONSIBLE FOR VERIFICATION (name, organization)
Chain-of-Custody Records and shipping documentation	Chain-of-Custody Records and shipping documentation will be reviewed by the laboratory upon receipt of samples for verification against the sample coolers they represent. The Chain-of-Custody Record will be signed by all parties who had custody of samples, with the exception of commercial carriers.	Internal	Dennis Catalano, WATS (or designee)
Field notes and sampling logs	All field notes and sampling logs will be reviewed internally and placed in the project file.	Internal	Jennifer Overvoorde, RMT
Laboratory data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal.	Internal	Dennis Catalano, WATS
Laboratory data	All final data packages will be verified for content upon receipt.	External	Greg Graf, ECCS (third-party data validator)

QAPP Worksheet #35
Validation (Step I) Process

STEP IIa/IIb	VALIDATION INPUT	DESCRIPTION	RESPONSIBLE FOR VERIFICATION (Name, Organization)
Step IIa	Sampling methods and procedures	Verify that the required analytical methods were used and that any deviations were noted.	Greg Graf, ECCS
Step IIa	Analytical methods	Verify that the required sampling methods were used and that any deviations were noted. The laboratory will verify that QC samples met the performance criteria, and that any deviations were noted on the sample narrative.	Dennis Catalano, WATS Greg Graf, RMT
Step IIb	Documentation of QAPP QC sample results	Establish that all QC samples required by the QAPP were collected and analyzed.	Greg Graf, ECCS
Step IIb	Project Quantitation Limits	Determine that the QAPP-required Quantitation Limits were achieved.	Greg Graf, ECCS
Step IIb	Validation report	Summarize data findings of verification and validation components included in the QAPP. Include comments on any qualified data, and define all qualifiers.	Greg Graf, ECCS

QAPP Worksheet #36
Validation (Steps IIa and IIb) Summary

STEPS 1Ia AND IIb	MATRIX	ANALYTICAL GROUP	DATA PURPOSE	CONCENTRATION LEVEL	VALIDATION CRITERIA	DATA VALIDATOR (title and organizational affiliation)
IIa and IIb	Aqueous/ Soil/ Sediment	PCBs, VOCs, PAHs	Site Investigation, Remedial Design, or Confirmation of Remediation	Low, medium, high	U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999), method criteria, QAPP criteria; and professional judgment	Greg Graf (ECCS)
IIa and IIb	Aqueous/ Soil/ Sediment	Metals	Site Investigation, Remedial Design, or Confirmation of Remediation	Low, medium, high	U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004), method criteria, QAPP criteria; and professional judgment	Greg Graf (ECCS)
IIa and IIb	Water	PCBs, VOCs, PAHs	Site Investigation, Remedial Design, Confirmation of Remediation, or Long Term Monitoring	Low, medium, high	U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999), method criteria, QAPP criteria; and professional judgment	Greg Graf (ECCS)
IIa and IIb	Water	Metals	Site Investigation, Remedial Design, Confirmation of Remediation, or Long Term Monitoring	Low, medium, high	U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004), method criteria, QAPP criteria; and professional judgment	Greg Graf (ECCS)
IIa and IIb	Oil	PCBs, Specific Gravity, & Viscosity	Site Investigation	Low, medium, high	U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999), method criteria, QAPP criteria; and professional judgment	Greg Graf (ECCS)
IIa and IIb	Aqueous/ Soil/ Sediment	General chemistry parameters	Confirmation of remediation	Low, medium, high	U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999), method criteria, QAPP criteria; and professional judgment	Greg Graf (ECCS)

QAPP Worksheet #37
Usability Assessment

Identify the personnel responsible for performing the usability assessment:

Environmental Chemistry Consulting Services, Inc. (ECCS), will perform the usability assessment for analytical data. Greg Graf is responsible for data validation at ECCS. The RMT Field Coordinator Is responsible for verification of field activities.

The RMT Project Manager, Project Scientists, and Laboratory Coordinator will determine if field and analytical data or datasets meet the requirements necessary for decision-making. The results of these measurements will be compared with the DQO requirements set forth in this QAPP. As data are evaluated, anomalies in the data or data gaps may become apparent to the data users. The DQOs will be satisfied if the data are sufficient (based on the quality and completeness of the data) to meet the project objectives.

Data that do not meet the data users' needs (if any) will be identified and appropriately qualified in the project database so that the decision-makers are aware of the limitations.

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

The Data Reviewer will review field notes and field Chain-of-Custody Records to determine that procedures specified in the FSP have been followed.

The RMT Laboratory Coordinator or designated Data Reviewer will conduct a systematic review of the data for compliance with the established QC criteria based on the spike, duplicate, and blank results provided by the laboratory. Data validation will determine whether the procedures specified in the FSP and in this QAPP were implemented, the DQOs specified in this QAPP were attained, the specified Quantitation Limits were achieved, and the sample holding times were met. An evaluation of data accuracy, precision, sensitivity, and completeness, based on method-specific criteria, will be performed according to the following guidance documents:

- National Functional Guidelines for Organic Data Review (U.S. EPA, 1999).

Method specifications provided in the U.S. EPA-approved methods and laboratory SOPs will be used as a guide for validating data obtained for all analytes listed in this QAPP.

All forms will be checked. All raw data, including chromatograms, quantitation reports, data system printouts, and mass spectra for samples, standards, performance evaluation mixtures, blanks, and laboratory spikes/laboratory duplicates or MS/MSDs will be reviewed. A portion (~10 percent) of calculations such as spike recoveries, calibration response factors, analyte quantitation, etc., will be randomly spot-checked.

QAPP Worksheet #37
Usability Assessment

The procedures used to evaluate data include the following:

- All technical holding times will be checked for inorganic and organic analyses.
- Instrument performance check sample results, and initial and continuing calibration results, will be evaluated.
- Data for all blanks, surrogate spikes, matrix spikes/matrix spike duplicates, laboratory control samples, cleanup standards, internal and external standards, target compound identification and quantitation, and system performance checks will be reviewed.
- Sample calculations will be checked.
- Field precision will be determined from blind field duplicate data.
- Completeness of the data package will be checked to determine if all samples and analyses required by the QAPP were processed, that the procedures specified in the QAPP were implemented, and that all deliverables specified in the QAPP are included.
- The Data Reviewer will identify any out-of-control data points and data omissions, and will interact with the laboratory to correct data deficiencies.
- Decisions to repeat sample collection and analyses may be made by the RMT Project Manager based on the extent of the deficiencies and their importance in the overall context of the project.
- The third-party data reviewer will assess the usability of results against the DQOs.

QAPP Worksheet #37 (*continued*)
Usability Assessment

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

The data quality indicator (DQIs) used to evaluate conformance with the project DQOs are presented below.

DQIs are generally defined in terms of the following six parameters;

1. Representativeness
2. Comparability
3. Completeness
4. Precision
5. Accuracy
6. Sensitivity

Each parameter is defined below. Specific objectives for the site actions are presented in other sections of this QAPP, as referenced below.

Representativeness

Representativeness is the degree to which sampling data accurately and precisely represent site conditions, and is dependent on sampling and analytical variability and the variability of environmental media at the site. Actions have been designed to assess the presence of chemical constituents at the time of sampling. The QAPP presents the rationale for sample quantities and location. This QAPP presents field sampling and laboratory analytical methodologies. Use of the prescribed field and laboratory analytical methods with associated holding times and preservation requirements is intended to provide representative data.

Comparability

Comparability is the degree of confidence with which one data set can be compared with another. Comparability between phases of the actions (if additional phases are required) will be maintained through consistent use of the sampling and analytical methodologies set forth in the QAPP, the established QA/QC procedures, and the use of appropriately trained personnel.

Completeness

Completeness is defined as a measure of the amount of valid data obtained from an event and/or investigation compared to the total amount that was obtained. This will be determined upon final assessment of the analytical results. Completeness of a field or laboratory data set will be calculated by comparing the number of valid sample results generated with the total number of results generated.

$$\text{Completeness} = \frac{\text{Number valid results}}{\text{Total number of results generated}} \times 100$$

As a general guideline, overall project completeness is expected to be at least 90 percent. The assessment of completeness will require professional judgment to determine data usability for intended purposes.

QAPP Worksheet #37 (*continued*)
Usability Assessment

Precision

Precision is a measure of the reproducibility of sample results. The goal is to maintain a level of analytical precision consistent with the objectives of the action. To maximize precision, sampling and analytical procedures will be followed. All work for the site actions will adhere to the established protocols presented in the QAPP. Checks for analytical precision will include the analysis of MS/MSDs, laboratory duplicates, and field duplicates. Checks for field measurement precision will include duplicate field measurements.

The precision of data will be measured by calculating the Relative Percent Difference (RPD) by the following equation:

$$RPD = \frac{(A-B)}{(A+B)/2} \times 100,$$

where

A = analytical result from one of two duplicate measurements,
B = analytical result from the second measurement.

Accuracy

Accuracy is a measure of how close a measured result is to the true value. Both field and analytical accuracy will be monitored through initial and continuing calibration of instruments. In addition, reference standards, MSs, blank spikes, and surrogate standards will be used to assess the accuracy of the analytical data.

Accuracy will be calculated in terms of percent recovery as follows:

$$\% \text{ Recovery} = \frac{A-X}{B} \times 100,$$

where

A = value measured in spiked sample or standard,

X = value measured in original sample,

B = true value of amount added to sample or true value of standard.

Sensitivity

Sensitivity is a quantitative measurement to determine if the analytical laboratory's procedures/methodologies and their associated MDLs can satisfy the project requirements as they relate to the project action limits. MDLs are updated annually by the laboratory.

QAPP Worksheet #37 (*continued*)
Usability Assessment

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:

The data validation report will address the following items:

- Overall quality and usability of the data
- Evaluation of QC data, including precision, accuracy, and completeness of the data
- Potential sample contamination due to blank contributions
- Assessment of laboratory and field records
- Actions regarding specific QC criteria exceedences

Laboratory-applied data qualifiers will be defined within the analytical data package received from the laboratory. The sample narrative will also detail quality control issues identified by the laboratory.

Data validation qualifiers that may be applied to the data include the following:

U The analyte/compound was analyzed for, but not detected. The associated value is the compound's Limit of Quantitation.

UJ The compound was not detected above the reported sample's Limit of Quantitation. However, the reported limit is approximate and may or may not represent the actual Limit of Quantitation.

J The compound was positively identified; however, the associated numerical value is an estimated concentration only.

R The sample results are rejected.

Attachment 1-1
Laboratory Accuracy and Precision Objectives

Table 1-1
Laboratory Accuracy and Precision Objectives
for PCBs and Total Phosphorus and TSS in Surface Water
Multi-Area QAPP
Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

ANALYTE	LCS/MATRIX SPIKE RECOVERY (% water)	LCS/MATRIX SPIKE RECOVERY (% soil)	LCS/MATRIX SPIKE RECOVERY (% free product)	RPD (% water)	RPD (% soil)	RPD (% free product)
Aroclor-1016	60-120	60-120	60-130	0-20	0-40	0-30
Aroclor-1260	60-120	60-120	60-130/41-62	0-20	0-40	0-30
Total phosphorus	90-110	--	--	0-20	--	--
TSS	85-115	--	--	0-20	--	--

Table 1-2
Laboratory Accuracy and Precision Objectives
For DRO/GRO in soil
Multi-Area QAPP
Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

ANALYTE	LCS/MATRIX SPIKE RECOVERY (% soil)
Gasoline	50-116
Diesel	61-145

Footnotes:

The most current in-house generated control limits will be used at the time of sample log-in.

Table 1-3
Laboratory Accuracy and Precision Objectives for Metals and Cyanide in Groundwater and Soil
Multi-Area QAPP - Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

ANALYTE[(1)]	LCS/MATRIX SPIKE RECOVERY (% water)	MATRIX SPIKE RECOVERY (%) (soil)	RPD (% water)	RPD (%) (soil)
Aluminum	85-115	70-130	0-20	0-40
Antimony	85-115	70-130	0-20	0-40
Arsenic	85-115	70-130	0-20	0-40
Barium	85-115	70-130	0-20	0-40
Beryllium	85-115	70-130	0-20	0-40
Cadmium	85-115	70-130	0-20	0-40
Calcium	85-115	70-130	0-20	0-40
Chromium	85-115	70-130	0-20	0-40
Chromium	85-115	70-130	0-20	0-40
Cobalt	85-115	70-130	0-20	0-40
Copper	85-115	70-130	0-20	0-40
Iron	85-115	70-130	0-20	0-40
Lead	85-115	70-130	0-20	0-40
Magnesium	85-115	70-130	0-20	0-40
Manganese	85-115	70-130	0-20	0-40
Mercury (total)	85-115	70-130	0-20	0-40
Mercury (Low-level)	71-125	NA	0-24	NA
Nickel	85-115	70-130	0-20	0-40
Potassium	85-115	70-130	0-20	0-40
Selenium	85-115	70-130	0-20	0-40
Silver	85-115	70-130	0-20	0-40
Sodium	85-115	70-130	0-20	0-40
Thallium	85-115	70-130	0-20	0-40
Vanadium	85-115	70-130	0-20	0-40
Zinc	85-115	70-130	0-20	0-40
Cyanide	75-125	75-125	0-20	0-20

Footnotes:
The most current in-house generated control limits will be used at the time of sample log-in.
NA = not applicable.
(1) All Metals and Cyanide will be analyzed by WATS except for low-level Mercury which will be analyzed by CAS.

Table 1-4
Laboratory Accuracy and Precision Objectives
for SVOCs in Groundwater and Soil
Multi-Area QAPP
Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

ANALYTE	LCS/MATRIX SPIKE RECOVERY (% water)	MATRIX SPIKE RECOVERY (%) (soil)	RPD (% water)	RPD (%) (soil)
2,2-oxybis(1-Chloropropane)	NS	NS	NA	NA
2,4,5-Trichlorophenol	NS	NS	NA	NA
2,4,6-Trichlorophenol	NS	NS	NA	NA
2,4-Dichlorophenol	NS	NS	NA	NA
2,4-Dimethylphenol	NS	NS	NA	NA
2,4-Dinitrophenol	NS	NS	NA	NA
2,4-Dinitrotoluene	62-125	70-112	0-38	0-38
2,6-Dinitrotoluene	NS	NS	NA	NA
2-Chlorophenol	71-100	56-97	0-40	0-40
2-Methylnaphthalene	NS	NS	NA	NA
2-Methylphenols	NS	NS	NA	NA
2-Nitroaniline	NS	NS	NA	NA
2-Nitrophenol	NS	NS	NA	NA
3,3-Dichlorobenzidine	NS	NS	NA	NA
3-Nitroaniline	NS	NS	NA	NA
4,6-Dinitro-2-methylphenol (2-methyl-4,6-dinitrophenol)	NS	NS	NA	NA
4-Bromophenyl-phenylether	NS	NS	NA	NA
4-Chloro-3-methylphenol	68-111	67-106	0-42	0-42
4-Chloroaniline	NS	NS	NA	NA
4-Chlorophenyl-phenylether	NS	NS	NA	NA
4-Methylphenols	NS	NS	NA	NA
4-Nitroaniline	NS	NS	NA	NA
4-Nitrophenol	52-117	53-124	0-50	0-50
Acenaphthene	70-104	67-105	0-31	0-31

Table 1-4
Laboratory Accuracy and Precision Objectives
for SVOCs in Groundwater and Soil
Multi-Area QAPP
Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

ANALYTE	LCS/MATRIX SPIKE RECOVERY (% water)	MATRIX SPIKE RECOVERY (%) (soil)	RPD (% water)	RPD (%) (soil)
Acenaphthylene	NS	NS	NA	NA
Anthracene	NS	NS	NA	NA
Benzaldehyde	NS	NS	NA	NA
Benzo(a)anthracene	NS	NS	NA	NA
Benzo(a)pyrene	NS	NS	NA	NA
Benzo(b)fluoranthene	NS	NS	NA	NA
Benzo(g,h,i)perylene	NS	NS	NA	NA
Benzo(k)fluoranthene	NS	NS	NA	NA
bis(2-Chloroethoxy)methane	NS	NS	NA	NA
bis(2-Chloroethyl)ether	NS	NS	NA	NA
bis(2-Ethylhexyl)phthalate	NS	NS	NA	NA
Butyl benzyl phthalate	NS	NS	NA	NA
Carbazole	NS	NS	NA	NA
Chrysene	NS	NS	NA	NA
Dibenzo(a,h)anthracene	NS	NS	NA	NA
Dibenzofuran	NS	NS	NA	NA
Diethyl phthalate	NS	NS	NA	NA
Dimethyl phthalate	NS	NS	NA	NA
Di-n-butylphthalate	NS	NS	NA	NA
Di-n-octyl phthalate	NS	NS	NA	NA
Fluoranthene	NS	NS	NA	NA
Fluorene	NS	NS	NA	NA
Hexachlorobenzene (C-66)	NS	NS	NA	NA
Hexachlorobutadiene (C-46)	NS	NS	NA	NA
Hexachlorocyclopentadiene (C-56)	NS	NS	NA	NA
Hexachloroethane	NS	NS	NA	NA

Table 1-4
Laboratory Accuracy and Precision Objectives
for SVOCs in Groundwater and Soil
Multi-Area QAPP
Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

ANALYTE	LCS/MATRIX SPIKE RECOVERY (% water)	MATRIX SPIKE RECOVERY (%) (soil)	RPD (% water)	RPD (%) (soil)
Indeno(1,2,3-cd)pyrene	NS	NS	NA	NA
Isophorone	NS	NS	NA	NA
Naphthalene	NS	NS	NA	NA
Nitrobenzene	NS	NS	NA	NA
n-Nitroso-di-n-propylamine	70-103	59-100	0-38	0-38
N-Nitrosodiphenylamine	NS	NS	NA	NA
Pentachlorophenol	63-109	66-114	0-50	0-50
Phenanthrene	NS	NS	NA	NA
Phenol	61-103	54-99	0-42	0-42
Pyrene	81-132	64-151	0-31	0-31

Footnotes:
The most current in-house generated control limits will be used at the time of sample log-in.
NS = not spiked.
NA = not applicable.

Table 1-5
Laboratory Accuracy and Precision Objectives for VOCs in Soil and Groundwater
Multi-Area QAPP - Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

ANALYTE	LCS/MATRIX SPIKE RECOVERY (% water)	LCS RECOVERY (% soil)	MATRIX SPIKE RECOVERY (% soil)	RPD (% water)	RPD (% soil)
1,1,1-Trichloroethane	NS	69-126	62-126	NA	40
1,1,2,2-Tetrachloroethane	NS	71-126	53-141	NA	40
1,1,2-Trichloroethane	NS	79-120	72-121	NA	40
1,1-Dichloroethane	NS	79-120	72-118	NA	40
1,1-Dichloroethylene	61-145	79-132	68-132	0-20	40
1,2,3-Trichlorobenzene	NS	65-117	64-119	NA	40
1,2,4-Trichlorobenzene	NS	68-110	65-116	NA	40
1,2-Dibromoethane (ethylene dibromide)	NS	74-121	72-119	NA	40
1,2-Dichlorobenzene	NS	80-112	77-109	NA	40
1,2-Dichloroethane	NS	67-129	66-123	NA	40
1,2-Dichloropropane	NS	81-117	74-117	NA	40
1,3-Dichlorobenzene	NS	80-112	75-111	NA	40
1,4-Dichlorobenzene	NS	80-113	73-111	NA	40
2-Butanone (MEK)	NS	70-117	69-124	NA	40
2-Hexanone	NS	66-118	56-138	NA	40
4-Methyl-2-pentanone (MIBK)	NS	68-115	62-126	NA	40
Acetone	NS	59-114	59-118	NA	40
Benzene	76-127	82-115	71-117	0-20	40
Bromochloromethane	NS	74-129	72-128	NA	40
Bromodichloromethane	NS	75-121	70-121	NA	40
Bromoform	NS	67-131	59-132	NA	40
Bromomethane	NS	23-121	10-126	NA	40
Carbon disulfide	NS	73-125	64-129	NA	40
Carbon tetrachloride	NS	60-129	56-128	NA	40
Chlorobenzene	75-130	82-116	74-117	0-20	40
Chloroethane	NS	57-125	58-130	NA	40
Chloroform	NS	66-124	65-118	NA	40

Table 1-5
Laboratory Accuracy and Precision Objectives for VOCs in Soil and Groundwater
Multi-Area QAPP - Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

ANALYTE	LCS/MATRIX SPIKE RECOVERY (% water)	LCS RECOVERY (% soil)	MATRIX SPIKE RECOVERY (% soil)	RPD (% water)	RPD (% soil)
Chloromethane	NS	49-124	48-134	NA	40
cis-1,2-Dichloroethylene	NS	86-125	73-129	NA	40
cis-1,3-Dichloropropene	NS	73-121	68-124	NA	40
Cyclohexane	NS	70-130	70-130	NA	40
Dibromochloromethane	NS	66-131	62-126	NA	40
Dibromochloropropane (1,2-Dibromo-3-chloropropane)	NS	58-119	52-134	NA	40
Dichlorodifluoromethane	NS	10-168	21-164	NA	40
Ethylbenzene	NS	80-121	72-124	NA	40
Isopropyl benzene	NS	75-108	53-124	NA	40
m,p-Xylenes	NS	82-118	71-127	NA	40
Methyl acetate	NS	70-130	70-130	NA	40
Methylcyclohexane (methyl phenol)	NS	70-130	70-130	NA	40
Methylene chloride	NS	69-125	69-117	NA	40
Methyl-tert-butyl ether (MTBE)	NS	65-120	68-119	NA	40
o-Xylene	NS	83-118	74-124	NA	40
Styrene	NS	89-122	77-128	NA	40
Tetrachloroethylene	NS	71-128	58-129	NA	40
Toluene	76-125	79-117	65-120	0-20	40
trans-1,2-Dichloroethylene	NS	79-118	70-118	NA	40
trans-1,3-Dichloropropene	NS	58-120	52-127	NA	40
Trichloroethylene	71-120	75-127	63-130	0-20	40
Trichlorofluoromethane	NS	57-126	53-122	NA	40
Vinyl chloride	NS	58-129	57-141	NA	40

Footnotes:
The most current in-house generated control limits will be used at the time of sample log-in.
NS = not spiked.
NA = not applicable.

Attachment 1-2
Surrogate Compound Recovery Limits

Table 2-1
PCB Surrogate Compound Recovery Limits
Multi-Area QAPP - Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

COMPOUND	% RECOVERY (water)	% RECOVERY (soil)	% RECOVERY (free product)
Tetrachloro-m-xylene	60-120	60-120	19-93
Decachlorobiphenyl	60-120	60-120	-

Table 2-2
DRO/GRO Surrogate Compound Recovery Limits
Multi-Area QAPP - Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

COMPOUND	% RECOVERY (soil)
Trifluorotoluene (GRO)	64-135
Bromofluorobenzene (GRO)	54-140
o-terphenyl (DRO)	72-130

Table 2-3
VOC Surrogate Compound Recovery Limits
Multi-Area QAPP - Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

COMPOUND	% RECOVERY (water)	% RECOVERY (soil)
1,2-Dichloroethane-d4	80-120	66-119
Toluene-d8	80-120	77-123
Dibromofluorobenzene	-	69-118
4-Bromofluorobenzene	80-120	58-135

Table 2-4
SVOC Surrogate Compound Recovery Limits
Multi-Area QAPP - Weyerhaeuser Company
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

COMPOUND	% RECOVERY (water & soil)
2-Fluorophenol	59-88
Phenol-d5	67-94
Nitrobenzene-d5	60-102
2-Fluorobiphenyl	65-99
2,4,6-Tribromophenol	48-100
Terphenyl-d14	64-150
2-Chlorophenol-d4	64-92
1,2-Dichlorobenzene-d4	50-96

Attachment 1-3
Sample Chain-of-Custody Record

State of Wisconsin
Department of Natural Resources

CHAIN OF CUSTODY RECORD
Form 4100-145 7-87

Sample Collector(s)	Title/Work Station	Telephone No. (include area code)
Property Owner	Property Address	Telephone No. (include area code)

Split Samples: Offered? ☐ Yes ☐ No (Check One)
Accepted? ☐ Yes ☐ No (Check One) Accepted By: _____
Signature

Field ID No.	Date	Time	Sample Type Comp	Sample Type Grab	Station Location Sample Description	Lab ID Number	No. of Containers	Comments

I hereby certify that I received, properly handled, and disposed of these samples as noted below:

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)

Disposition of Unused Portion of Sample:

Dispose _____ Retain for _____ days

Return _____ Other _____

Attachment 1-4
Laboratory Quality Assurance Manual

Weyerhaeuser

WEYERHAEUSER
ANALYSIS AND TESTING SERVICES
WATS Quality Assurance Manual

Weyerhaeuser Company
WTC 2F25
P.O. Box 9777
Federal Way, Washington 98063-9777

Effective Date: October 31, 2006

The policies and practices delineated in this document are fully supported by the management and staff members of Weyerhaeuser Analysis and Testing Services (WATS). These policies and practices are to be adhered to by everyone working in WATS. This commitment is made by the Laboratory Manager and the staff, as stipulated in Section 2.5.

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WATS Quality Assurance Manual

October 31, 2006

Quality Manual Title Page

Table of Contents
Revision History
Quality Policy Statement
Forward-Introduction

Chapter	Process
1	Quality Management Process
2	Resource Management Process
3	Product Design Process
4	Purchasing Process
5	Production and Service Processes
6	Product Protection Process
7	Customer Assessment -Communications Processes
8	Internal Communications Process
9	Document Control Process
10	Record Keeping Process
11	Planning Process
12	Training Process
13	Internal Audit Process
14	Management Review Process
15	Monitoring and Measuring Process
16	Nonconformance Management Process
17	Continual Improvement Process
Appendix	Acroymns
Appendix	WATS Process Flow

Index

Weyerhaeuser Analysis and Testing Services
WATS
Quality Assurance Manual
October 31, 2006

Revision History

.

A&T QUALITY ASSURANCE GUIDELINES AND POLICY

June 7, 2002
Effective Date

August 13, 2003
Reissue Date
Reissued with minor typographical corrections.

September 30, 2003
Reissue Date
"Weyerhaeuser Analysis and Testing Services Quality Assurance Guidelines and Policy August 13, 2003 version" was revised. Changes were made to reflect the organizational changes in the Quality Assurance Advisor role. Section 2.2.7 was rewritten. Section 2 changes also include minor grammatical changes to other roles. Sections 5.5, 5.6, 5.7, and 5.8 were extensively rewritten to reflect changes made to the procedures for document control, nonconformances, corrective action, preventive actions, and complaints. Minor typos and grammatical changes were made throughout the document

WATS Quality Assurance Manual

December 13, 2004
Rewritten Date, Draft Proposal Presented
The original quality document *A&T QUALITY ASSURANCE GUIDELINES AND POLICY* was totally revised and two documents were created, *WATS Quality Assurance Manual* and *WATS Laboratory Policies and Guidelines*.

July 14, 2005Effective Date
Weyerhaeuser Analysis and Testing Services *WATS Quality Assurance Manual* was proposed on December 13, 2004 and became effective July 14, 2005.

Chapter	Revision History	Author	WATS Quality Assurance Team
Comments	WATS Quality Assurance Manual	Date	October 31, 2006 revision

October 31, 2006 Effective Date

An extensive review and rewrite of the *WATS Quality Assurance Manual* was undertaken in December, 2005 to more accurately reflect the processes and policies of WATS.

December 5, 2007

The 2007 QMI external audit identified the WATS Process Flow Chart in the WATS Quality Manual as a system weakness (ISO 5.4.2) because it was not related to the WATS Processes. The Flow Chart was representative of the Sample Management process and renamed Process Map for Clients Samples-Requests.

January, 2008

Process Flow diagram replaced the WATS Process Flow Chart that will become a Sample Management tool.

The policies and practices delineated in this document are fully supported by the management and staff members of Weyerhaeuser Analysis and Testing Services (WATS). These policies and practices are to be adhered to by everyone working in WATS.

Chapter	Revision History	Author	WATS Quality Assurance Team
Comments	WATS Quality Assurance Manual	Date	October 31, 2006 revision

Quality Policy Statement (ISO 5.3)

WATS is committed to provide competitive technical services in a safe, timely, accurate, ethical, and cost-efficient manner. WATS is also committed to achieve customer satisfaction at all times.

WATS continually improves the effectiveness of the quality management system (QMS) and sets quality objectives that strive to ensure that there is an individual, team, and department commitment for improvement. Moreover, WATS is committed to upgrade the capabilities of its technical and administrative staff through formal training and continuous technical quality awareness.

WATS management is committed to maintaining its quality system to comply with the ISO Standard 9001:2000 and to introduce its staff to its quality policy and quality system. It is the responsibility of the staff, however, to familiarize themselves and comply with the policies and procedures of the quality system adopted. This system is contained in a primary Quality Manual for the whole of WATS and a set of secondary quality manuals that include laboratory guidelines and procedures.

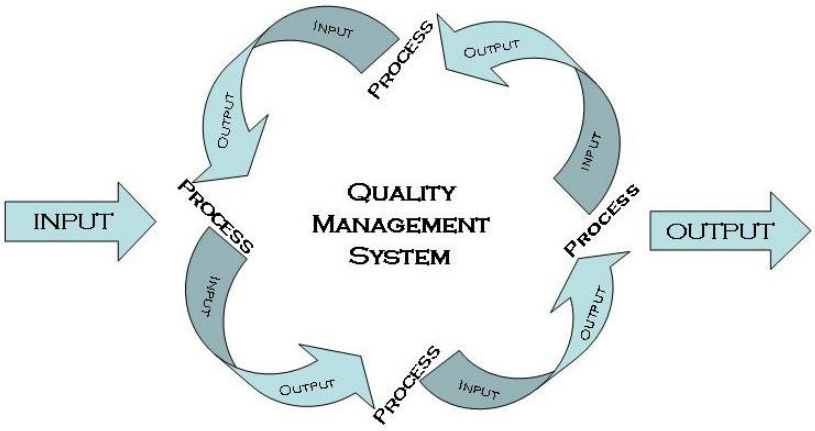
WATS (Weyerhaeuser Analysis & Testing Services) operates within one Quality Management System (QMS). This system covers all testing, research, and development done by Cellulose Properties (formerly Pulp and Paper and currently part of Cellulose Fibers Technology Properties (CFTP)), Analytical, and Microstructure. (Components of WATS Pulp and Paper testing are encompassed by these three labs.) The labs for Absorbent Products, Containerboard, and Crosslinked are part of Cellulose Fibers Technology Properties (CFTP) and may become part of the QMS if it is determined by the Operations Managers that there is a benefit to including them. Only products and materials produced by WATS Pulp and Paper Testing are covered by the ISO 9001:2000 standard. Part 7.3 of ISO 9001:2000 is excluded from the WATS QMS. Methods used to produce test results for clients requiring WATS Pulp and Paper Testing are predetermined by our clients.

Forward

ISO organized the ISO 9001 standard to create a logical structure compatible with the ISO 14001 environmental management standard. The standard is customer-oriented and it expects WATS to communicate with customers and to measure and monitor customer satisfaction. The WATS quality management system complies with the ISO 9001 standards.

The new standard tells you what to do, not how to do it. The standard also emphasizes the need to make improvements. ISO 9001 requires us to evaluate the effectiveness and suitability of our quality management system, and to identify and implement systemic improvements.

The ISO standard uses a process approach to quality management. A quality management system is a single large process that uses many inputs to generate many outputs. This large process is, in turn, made up of many smaller processes. Each process uses inputs to generate outputs, and all of these processes are interconnected using these input-output relationships.



The ISO 9001:2000 Quality Management System is made up of processes and procedures. A quality management system that meets the ISO 9001:2000 standard has designed, documented, and implemented the processes identified in its quality management system. These processes must be supported, monitored, controlled, and improved.

The required procedures from the ISO standard are:

- Document Control (clause 4.2.3)
- Control of Quality Records (clause 4.2.4)
- Internal Audit (clause 8.2.2)
- Control of Nonconformity (clause 8.3)
- Corrective Action (clause 8.5.2)
- Preventive Action (clause 8.5.3)

.

ISO 9000:2000 has also introduced mandatory requirements:

- Meet legal requirements (5.1, 5.2).
- Meet regulatory requirements (5.1, 5.2).
- Meet customer needs and expectations (5.2).
- Support internal communication (5.5.3).
- Evaluate the effectiveness of training (6.2.2).
- Provide quality facilities-infrastructure (6.3).
- Provide a quality work environment (6.4).
- Identify customer needs and expectations (5.2, 7.2.1).
- Communicate with customers (7.2.3).
- Measure and monitor customer satisfaction (8.2.1).
- Measure and monitor realization of processes (8.2.3).
- Evaluate the effectiveness and suitability of quality system (8.4).
- Identify quality management system improvements (5.6.3, 8.2.2).
- Improve quality management system (5.1, 8.5).

For more detail, please see the associated ISO 9001:2000 clauses (in parenthesis).

The WATS Quality Assurance Manual is written to reflect and define the processes used to create its Quality Management System. The ISO standard recognizes that there may be twenty processes necessary for an effective quality management system (with the entire QMS being thought of as the 21st process).

- Quality Management Process
- Resource Management Process
- Regulatory Research Process
- Product Design Process
- Purchasing Process
- Production Process
- Service Provision Process
- Product Protection Process
- Customer Needs Assessment Process
- Customer Communications Process
- Internal Communications Process
- Document Control Process
- Record Keeping Process
- Planning Process
- Training Process
- Internal Audit Process
- Management Review Process
- Monitoring and Measuring Process
- Nonconformance Management Process
- Continual Improvement Process

Chapter	Forward	Author	WATS Quality Assurance Team
Document	WATS Quality Assurance Manual	Date	October 31, 2006 revision

1.0 **Quality Management Process** (ISO 4.1)

1.1 Quality is achieved by ensuring that adequate and accepted quality processes and procedures are used throughout WATS, and by establishing a means to monitor and assess performance on these activities.

1.2 Weyerhaeuser Analysis and Testing Services (WATS) meets and complies with the requirements of organizations or rules, such as ISO 9001, laboratory certification, or regulations, under which it is certified, accredited, or obligated to follow.

1.3 The ISO standards are an International certification program that ensures that a product vendor meets minimal standards for adherence to a corporate-wide manufacturing and services quality process (ISO 9002) and that such quality standards extend to the product design and development process (ISO 9001).

Our implementation of the ISO 9001 quality system includes only products or materials produced by WATS' Pulp and Paper Testing organization (Components derived from Cellulose Properties, Analytical, and Microstructure). It is not meant to be inclusive of research, developmental processes in support of research, or environmental testing. Absorbent Products, Containerboard, and Crosslinked laboratories may be included within the scope of this document at the discretion of the Operations Managers. Inclusion of these labs in the ISO 9001 quality system will be determined by the Operations Managers.

Part 7.3 of ISO 9001.2000 is excluded from the ISO system. WATS Pulp and Paper Testing (Components derived from Cellulose Properties, Analytical, and Microstructure) does not develop testing procedures. Test results are derived from methodologies stipulated and approved of by its customers. These processes are included within the scope of the quality management system, not under the registrations of our ISO 9001 certifications.

> **Exceptions:** Certain aspects of this plan may not apply to certain laboratory groups and test procedures that are not involved in regulatory required testing or testing falling outside the scope of ISO. In such cases, the Quality Assurance/Quality Control/ISO Team Members may approve deviations, but this must be documented. This is generally done by an SOP, but other processes may be approved.

1.4 Weyerhaeuser Analysis and Testing Services (WATS) recognizes that Quality Management (QM) requires individual, team, and unit commitment. Everyone is expected to maintain a commitment to these practices and to the quality of our testing services.

1.5 Weyerhaeuser Analysis and Testing Services (WATS) reports administratively to Weyerhaeuser R&D, a division of the Weyerhaeuser Company. R&D reports directly to the Weyerhaeuser's senior vice-president of technology.

1.6 WATS is an amalgamation of laboratories. WATS is organized in a manner that operationally crosses these laboratory boundaries. The laboratories for Analytical, Cellulose Properties (CP)

(Part of Cellulose Fibers Technology. Formerly Pulp and Paper Testing.), and Microstructure consist of smaller laboratories that include:

- Weyerhaeuser Environmental Laboratories, they must meet environmental reporting requirements that vary between regulations and jurisdictions.
- Weyerhaeuser Cellulose Properties (CP) (formerly Pulp and Paper Testing) who must meet requirements stipulated by its customers and operate its quality system in accordance with ISO 9001:2000.
- Weyerhaeuser Research Laboratories that support Corporate Research and Development.
- Weyerhaeuser Absorbent Products, Crosslinked, and Containerboard Laboratories supporting the Cellulose Fibers Technology (CFT) business and its customers.

1.7 WATS is committed to producing a product satisfactory to the needs of its clients in a safe, timely, accurate, ethical, and cost efficient manner by insuring that there are sufficient quality assurance (QA) activities conducted within WATS to ensure that all analytical and testing data generated and processed will:

- be scientifically valid
- have known precision and accuracy
- be acceptably complete
- be representative
- be comparable
- be legally defensible.

1.8 WATS improves the effectiveness of the quality system by establishing yearly quality goals. These goals are established and reviewed at the periodic managerial meetings and the formal management reviews. Goals are communicated to the department through e-mail distribution, company web page and staff meetings.

2.0 **Resource Management Process** (ISO 5.5.1, 5.5.2)

2.1 Management is committed to ensuring the availability of resources needed to implement, maintain, and improve WATS' quality management system. The goal of the WATS quality management system is to ensure customer satisfaction by meeting customer requirements.

2.2 Management is responsible for making people aware of how their work affects product quality. Maintaining this high level of quality ensures customer satisfaction with our product (customer data).

2.3 Performance management plays an important role in personnel development and the company's salary planning process. Tools and information to create individual development plans and help with career goals are available through the Weyerhaeuser Company website located within the "My Guide" section on myROOTS. Resources to support employees that need to take time off or experience a work-related event, such as a transfer to a new location, are available through the web site.

2.4 Laboratory managers supply their employees with sources to obtain mentor support and information, and education and training resources. The laboratory managers initiate an annual performance management process that includes goal setting, Individual Development Plans (IDP) and performance reviews.

2.5 Roles and Responsibilities

2.5.1 PEOPLE LEAD (Management Representative)

- Safety
    - Review incidents for unit with help from the Safety Committee representative
    - Role model safety through committed leadership and by recognizing and managing risks in appropriate work areas
- Communication
    - Establish and maintain effective working relationships with coworkers, clients, organizations across the company, key business partners and external organizations
    - Interface between WATS and other leaders within the company
    - Company-wide issues
    - Communicate relative company-wide, corporate R&D, and WATS information to Operations Managers or to WATS employees at staff meetings
- Role
    - Salary planning (responsible) and PARS (responsible)
    - Strategic Direction of the Laboratory

- ESRs (Employee Staffing Requests)
- Budget and planning (responsible)

- Goals

  - Helps Operations Managers with setting team's Individual Development Plans and goals

2.5.2 OPERATIONS MANAGER (Laboratory Managers)

- Safety

  - Role model safety through committed leadership and by recognizing and managing risks in appropriate work areas (i.e. - RADAR).

- Communication

  - Provide management of the day-to-day operation of the laboratory
  - Establish and maintain effective working relationships with coworkers, clients, organizations across the company, key business partners and external organizations
  - Communications:
    1) Company-wide, CR&D, and WATS info to staff
    2) Keep People Lead informed of issues
    3) Open 2-way between Technical Specialist/Project Managers/Operations Managers with opportunity for input prior to decisions that affect the whole lab
  - Direct link with other organizations in the company
  - Final decision for flow-to-work issues and conflicts (with Technical Specialist and analyst input)
  - Coordinate with other Operations Managers in WATS

- Role

  - Salary planning (execution) and PARS (Performance Appraisal Reviews) execution with 360º input
  - Individual Development Plans/goals
  - Recognition and celebrations
  - Input to ESRs (Employee Staffing Requests)
  - Budget and planning (execution)
  - Performance Issues (responsible and execution)
  - Career development (responsible and execution)
  - Training and education of staff (responsible)
  - Management of temporary staffing
  - Vacation and attendance issues
  - Monitor overall day-to-day operation of the laboratory

- Fill in as needed for emergencies in areas of responsibility

- Goals

  - Reviews Individual Development Plans and goals on bi-annual basis with staff

2.5.3 PROJECT MANAGER

- Safety

  - Role model safety through committed leadership and by recognizing and managing risks in appropriate work areas (i.e. - RADAR)

- Communication

  - Act as the primary interface between WATS and its customers
  - Ensure projects are coordinated and completed in a manner that meets the customer's needs
  - Establish and maintain effective working relationships with coworkers, clients, organizations across the company, key business partners and external organizations
  - Customer liaison
  - Communicates with Technical Specialists about status of work, technical issues, workload/flow-to-work, and assigning LIMS test codes
  - Works with Technical Specialists and Operations Managers to assure flow-to-work needs are surfaced & resolved
  - Knowledge of other project manager areas
  - Build professional relationships between lab and assigned customers
  - Monitor status of work
  - Coordinate projects with Technical Specialists

- Role

  - Obtain impending work load information, define work plan, get customer project number, estimate/price setting
  - Estimate date of receipt
  - Request new customers, component lists, or test schedules from LIMS administrator
  - Communicate project info to lab
  - As SMO receives samples, PM's will:
    - Assess samples and assign tests and due date with Technical Specialists to meet data quality objectives (DQO) (responsible)
      1. Reconcile discrepancies of samples, paperwork, and work plans (responsible)
      2. Ensure appropriate bottles and volumes available for testing (responsible - delegate upon receipt when necessary)

3. Coordinate splitting and prep of samples
4. Handle holding time issues once notified by SMO (responsible)

- Review SR, approve and release to appropriate labs
- Ensure paperwork is filed (responsible but some of this could be delegated to the appropriate Technical Specialists)
- Review final report to ensure it is complete
- Distribute final report. Complete and approve SR (responsible but some should be delegated to Technical Specialist)
- Cancel SRs
- Ensure disposal of hazardous samples. Stipulating unique disposal time & archives (responsible)
- Knowledge of other project manager areas
- Validate billing information
- Fill in labs or SMO for emergencies

- Goals

  - Maintain own Individual Development Plans and goals with guidance from Operations Manager

2.5.4 TECHNICAL SPECIALIST

- Safety

  - Take personal responsibility for working injury free and role model safety through committed leadership and by recognizing and managing risks in appropriate work areas (i.e. - RADAR).
  - Insure complete mitigation of hazards associated with all lab processes and testing in areas of responsibility using the R&D Hazard Assessment process. Effectively communicate hazards to lab personnel.

- Communication

  - Focus on delivering quality product and technical expertise to the customer while keeping current with their technology specialty
  - Establish and maintain effective working relationships with coworkers, clients, organizations across the company, key business partners and external organizations
  - Serve as a technical expert in a specified field within the lab, including interfacing directly with clients for method development, answering questions on analyses, and testing needs
  - Communicate with Project Manager about status of work, technical issues, workload/flow-to-work, and assigning LIMS test codes
  - Establish priorities to meet due dates and coordinate the work within area
  - Coordinate sample splitting and prep with Project Managers and other labs

- Role

    - Generate data, reports, and QC review within specialty (LIMS and hard copy)
    - Stay current on advancements in technology within their technical specialty
    - Method development (responsible – Technical Specialist should interface directly with customers)
    - Support the technical training of technicians
    - Support the analysts and technicians as needed in actual testing
    - Delegate tasks to analysts or technicians as necessary
    - Recommend capital investments to the Operations Manager for their area of specialty
    - Ensure proper disposal of hazardous materials
    - Sign payroll time slips for contract workers
    - Review routine SRs delegated by Project Manager, approve and release to appropriate labs
    - Ensure data package is complete
    - Review final report to ensure it is complete and correct. Distribute final routine reports as delegated by Project Managers. Complete and approve SR.
    - Cancel routine SRs that have been delegated
    - Maintain QA /QC protocol for the labs with guidance from QA /QC advisor
    - Updating and writing SOPs and methods
    - Assist OM with managing temporary staff activities

- Goals

    - Maintain own Individual Development Plans (IDP) and goals with guidance from Operation Manager

2.5.5 SAMPLE MANAGEMENT (SMO)

- Safety

    - Take personal responsibility for working injury free and role model safety through committed leadership and by recognizing and managing risks in appropriate work areas (i.e. - RADAR).
    - Communicate any hazards (chemical-related and other) to the labs as samples arrive for processing.

- Communication

    - Support project managers (and labs) by receiving and distributing samples and appropriate paperwork
    - Establish and maintain effective working relationships with coworkers, clients, organizations across the company, key business partners and external organizations

- Interfacing directly with clients by answering questions on analyses, and testing needs
- Communicate with Project Manager about status of incoming samples, technical issues, workload/flow-to-work, and assigning LIMS test codes

- Role

  - Establish priorities to meet sample log-in timelines and coordinate the work within SMO area
  - Coordinate sample splitting and prep with Project Managers and other labs
  - Keep Project Managers current on status of work. Communicate late data and expected hang-ups / delays in a timely fashion
  - Work with Operations Managers and Project Managers to assure flow-to-work needs are surfaced & resolved
  - Bottle prep
  - Receive samples and identify hazards associated with any incoming samples per client paperwork
  - Enter sample/project info into LIMS
  - Assign SR numbers
  - Manage storage facilities
  - Disposal of routine samples plus disposal of hazardous samples with direction of Project Manager or Technical Specialist with appropriate documentation
  - DOT shipping - bottle orders and samples to other labs

- Goals

  - Maintain own Individual Development Plans (IDP) and goals with guidance from Operations Manager

2.5.6 ANALYSTS / TECHNICIANS

- Safety

  - Role model safety by recognizing and managing risks in appropriate work areas (i.e. - RADAR).

- Communication

  - Establish and maintain effective working relationships with coworkers, clients, organizations across the company, key business partners and external organizations
  - Establish priorities to meet due dates with the help of Technical Specialists / Project Managers
  - Alert Technical Specialists / Project Managers to issues that affect their ability to perform their duties including status of work, workload/flow-to-work needs

- Get input from Technical Specialist / Project Manager regarding proper disposal of hazardous materials

- Role

  - Focus on creating quality data to meet the clients' needs in a safe, timely, accurate and ethical manner
  - Support Technical Specialist in assigned duties
  - Enter data into the LIMS
  - Review data within areas trained (LIMS and hard copy)
  - Get input from Technical Specialists / Project Managers regarding proper disposal of hazardous materials
  - Maintain instrumentation with help of Technical Specialist
  - Keep supplies at a level to keep lab running smoothly (with approval of Technical Specialist where appropriate)
  - Lab housekeeping
  - Flow to work

- Goals

  - Maintain own Individual Development Plans (IDP) and goals with guidance from Operations Manager

2.5.7 LIMS / ADMIN

- Safety

  - Role model safety by recognizing and managing risks in appropriate work areas (i.e. - RADAR).

- Communication

  - Focus on supporting the lab staff to keep operations running smoothly
  - Provide electronic tools necessary to help the unit meet its goals (LIMS)
  - Establish and maintain effective working relationships with coworkers, clients, organizations across the company, key business partners and external organizations
  - Alert Technical Specialists / Project Managers / Operation Manager to issues that affect their ability to perform their duties
  - Keep supplies at a level to keep lab running smoothly (with approval of TS where appropriate)

- Role

  - Admin/ lab support where appropriate

- LIMS creation, operation and maintenance
- Support of staff through archival of data, ordering supplies, maintaining operations, etc.
- Fill in labs or SMO for emergencies

- Goals

  - Maintain own Individual Development Plans (IDP) and goals with guidance from Operations Manager

2.5.8 QUALITY ASSURANCE/ QUALITY CONTROL/ ISO TEAM MEMBERS

Everyone in WATS shares responsibility for quality assurance and is responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function.

- Safety

  - Role model safety through committed leadership and by recognizing and managing risks in appropriate work areas (i.e. - RADAR).

- Tasks:

  - Identifying quality problems within the processes
  - Initiating, recommending, or providing solutions to quality problems
  - Verifying effective implementation of the quality programs

- Communication

  - Quality Assurance/Quality Control/ISO Team Members report to the People Lead and advise the Technical Specialists, Project Managers, and Operations Managers, providing a focus for QA activities within WATS. QA/QC activities are independent from the laboratory operations.
  - Work with People Lead (Management Representative) and Operations Managers (Laboratory Managers) to meet their responsibilities of:
    1. communicating to the organization the importance of meeting customer and regulatory requirements
    2. establishing the quality policy
    3. ensuring quality objectives are established
    4. ensuring that the integrity of the quality system is maintained when changes to the quality system are planned and executed
    5. Work with Operations Managers to ensure that processes needed for the quality management system are established, implemented, and maintained
  - Work with Operations Managers to ensure the quality of all data reported by the laboratory

- Role

    - Work with teams to implement quality objectives
    - Oversee and/or review quality control data
    - Evaluate data objectively and perform assessments without outside (e.g. managerial) influence
    - Have a general knowledge of the analytical and testing methods for which data review is performed
    - Review all standard operating procedures (SOP), testing methods (TM), and other required operational documents
    - Approve and document test procedures that are not involved in regulatory required testing or testing that fall outside the scope of ISO (generally done by an SOP)
    - Identify quality problems, initiating, recommending, or providing solutions to quality problems and verifying effective implementation of the quality programs
    - Act as technical advisors, summarizing and reporting QA/QC performance notifying management of deficiencies in the quality system and monitoring corrective action
    - Develop, maintain, and interpret the QA/QC manual and any secondary procedures
    - Schedule, or conduct, QA/QC internal audits; as well as scheduling external ISO audits
    - Insure that WATS participates, when available, in inter-laboratory comparisons and proficiency testing programs. This includes round-robin programs, such as those specified by regulations, APG and other performance sample programs, certification activities (such as Washington State Dept. of Ecology/NELAP/ISO), and blind or reference sample analyses.

- Goals

    - Maintain own Individual Development Plans and goals with guidance from Operations Manager

Relevant SOPs, Test Methods, and Documents:
OP SMO

3.0 **Product Design Process** (ISO 4.1, 7.1)

3.1 WATS provides analytical testing services to its clients. The results of these services are test data in the form of a report, which is WATS' product. The product design process means offering products that enhance our customers' processes. Simply "giving customers what they want," while fundamental to customer satisfaction, is rarely enough. We must be able to give customers the right products in the most resource-effective manner, without sacrificing quality or service. Effective communication, management of change and process planning are essential ingredients for an effective product development process.

3.2 Project managers following the process shown in the WATS process flow chart are responsible for distributing data in the form of reports that are sent to the customer. (See appendix for WATS Process Flow Chart.)

3.3 Managing the process for distributing information to the customer insures that the highest level of confidence in test results is maintained. Customer feedback is routinely sought by the laboratory to determine if the customers' needs are being met and to actively solicit information to insure continual improvement.

3.4 Discussions and action items on improvements to the "product" for the customers are included in the management review process.

4.0 **Purchasing Process** (ISO 7.4)

4.1 The process for purchasing is detailed in the OQ PURCHASE procedure. WATS administrative assistants keep a list of approved vendors.

4.2 The WATS' purchasing process is aligned with the company's Suppliers Core Policy, Supply Chain Management Functional Policy, Corporate Procurement & Supply Management (P&SM) and HQ Region Chemical Management. The purchasing process insures that quality parts and supplies are delivered on time, at the agreed cost, and to our specification to ensure that our product will be delivered on time, at the agreed cost, to our customer's specification.

4.3 WATS administrative assistants purchase outside services and supplies for WATS staff.

NOTE: Exceptions are major equipment, which must go through the Company's "PACE" process.

4.4 WATS' staff is responsible for identifying problems in vendor dealings or with purchased items or services. These problems must be brought to the attention of the administrative person responsible for purchasing, the affected area's operations manager and the quality advisor. Problems include, but are not limited to, timely delivery, quality of the product or services, and cost. The operations manager is responsible for assessing the problem and determining a course of action, which is documented on the Supplier Feedback/Issue Resolution form found on the Purchasing webpage. Resulting activities are documented and shared with the administrative person doing the purchasing.

4.5 Effective purchasing results in reduced costs by helping eliminate wasted effort and material due to purchasing materials that do not fulfill our specifications. It also results in increased customer satisfaction from getting expected services.

Relevant SOPs, Test Methods, and Documents:
OQ PURCHASE

5.0 Service and Production Processes (ISO 4.1, 7.1)

5.1 Service provision is a process that transforms inputs into outputs and creates benefits by facilitating a change in the customers' physical possessions, or a change in their intangible assets. Quality assurance means to identify what the customer wants, to analyze if these requirements can be satisfied and controlled in the production process, and, finally, to check the product (data) against the customers' requirements. WATS' continuing efforts and activities towards ongoing improvements of the service provision process are demonstrated in the services available to its customers.

5.2 Within WATS, the following roles have been developed to satisfy these processes:

5.2.1 The Laboratory Manager has overall responsibility for WATS including ensuring the production and quality of all data reported by the laboratory.

5.2.2 The Operations Managers act as the management representative for their respective areas. They have responsibility and authority to promote an awareness of customer requirements throughout the organization. The Operations Managers work with all personnel to assure proper operations of the laboratory on a daily basis to ensure the production and quality of all data reported by the laboratory. (An Operations Manager may also, functionally, be a Project Manager or Technical Specialist.) They communicate to the organization the importance of meeting customer and regulatory requirements.

5.2.3 The Project Managers, in cooperation with the Technical Specialists and Operations Managers, jointly manage the workflow within the laboratory. They act as the primary interface between WATS and their customers, working cooperatively to make sure that their needs are understood and met.

5.2.4 The Technical Specialist is the link between the laboratory and customers on technical issues as well as a link between the laboratory and the scientific community both internal and external to Weyerhaeuser Company. The Technical Specialist is responsible for delivering a quality product to the customer.

5.3 The customer determines product (data) specifications. The processes to generate a product to meet the customers' specifications are located within test methods and SOPs. Products (data) not meeting specifications are identified, isolated until the problems are solved, and then modified before they are approved as accepted products. Potential problems are identified and the related actions taken in the process during production are documented following the SOPs for preventive/corrective actions and nonconformances.

5.4 Outputs of managerial review include action items to improve the product to satisfy customer requirements. To prevent the occurrence of, eliminate the cause of, and address the effect of nonconformities, customers of WATS can initiate corrective actions. The customers may contact any WATS team member to generate Corrective Action Reports (CAR).

5.5 Proprietary concerns on the part of the corporation's customers may limit the exchange of information within the corporation's business units. Work for customers external to Weyerhaeuser, and not associated with Weyerhaeuser (such as by subcontracting) is confidential and not to be shared with anyone other than those specific customers without their written permission. However, we are an internal corporate laboratory; we safeguard information but do not take exceptional measures beyond our normal procedures used to process data. External clients should be made aware of this in writing.

5.6 Data that is attorney-client privileged may involve revealing information only to a specified attorney. This can include all raw data, any reports, unused sample, etc., which may be required to be given to those attorneys. In such instances, data and information will not be revealed to any persons, including Weyerhaeuser employees, except as necessary to execute the work.

5.7 Quality records are stored as documentation both for internal audits as well as for customers following the standard operating procedures for record storage. The computerized system that these quality records are stored in electronically is ensured data security through regular backups and other system administration utilities. The computerized quality data is stored in a system, which is open, standardized and flexible, and fully implemented by being based on an SQL relational database as documented in the Outsourced Services Manual.

5.7.1 Periodically, collected information bearing on the quality system is assembled and evaluated by the quality advisor. Such information is used for establishing the state of the quality system in regards to suitability and effectiveness. Information may come from customer feedback, product conformance, corrective/preventative actions, complaints, and nonconformances. Typically, this information is presented during the management review.

5.7.2 The ISO standard requires documentation of the statistical techniques (mean, maximum, minimum and standard deviation) used in the production process and created on reports and graphics presented. These statistical techniques are documented in the Laboratory Policies and Guidelines Manual (LP&G).

See appendix for the WATS Process Flow Chart.

Relevant SOPs, Test Methods, and Documents:
Outsourced Services Manual
Laboratory Policies and Guidelines Manual (LP&G)
OQ CORRACT
OQ PREVENTACT
OQ NONCONFORM

6.0 **Product Protection Process** (ISO 7.5.5)

6.1 WATS has established and implemented procedures for protecting the integrity and confidentiality of data. There are processes for data entry or capture, data storage, data transmission, data processing, data security, and data access.

6.2 Employees are held to the highest standard of ethical conduct. The Weyerhaeuser Company and the WATS department both value integrity. All personnel will behave in an honest and responsible manner and conduct themselves accordingly. All measurements and reporting will be done objectively. Employees are trained on ethical and legal responsibilities before beginning employment. This training is documented and employees must agree to perform all activities in an ethical manner.

6.3 The recorded date, time of collection, preparation, and testing must match the actual date and time that the action was performed. These records shall include the identity of personnel involved in (sub) sampling, preparation, calibration, testing, and checking of results. This identification is normally done by initials, signature, or electronic identification in the Laboratory Information Management System, LIMS.

All original data shall be recorded directly, promptly, and legibly in permanent (indelible) ink (except those that are generated by automated data collection systems). Such information shall be identifiable to a specific task. (Blue ink color is preferred in order to differentiate original data from copier data.)

6.4 Reports generated or stored on personal computers or servers, excepting the LIMS, shall be kept in hard copy form, or in write-protected files. Files on computers are kept until archived and stored on a disc with the administrative personnel or Computer Report Specialist. (The periodic archival process is done through Weyerhaeuser contracted IT.) For PCs with critical data, this is done by the owner with separate discs or by saving to the server.) All hardware and software necessary for the historical reconstruction or retrieval of data and other necessary information must be maintained by the laboratory. The LIMS is designed so no original data can be lost.

6.5 Certifications, analyst training documents and other documents (or copies of them) relating to the quality of the work in the laboratory are kept in a file by the administrative personnel. Retired files required to be retained are filed as SRs in Archived Service Requests (ASR). (Previous retired files were filed in ORB (original record book) and BARF (boxed archived record file).) ASRs and LIMS data are stored according to the Company's record retention policies.

Records are securely stored either under control of the laboratory or at Weyerhaeuser's records retention facilities. These records are protected from damage, deterioration, and loss. They are easily retrievable by the laboratory's tracking system.

Records stored at a Weyerhaeuser records retention facility may be destroyed in accordance with the retention facility's policies and procedures. At client (or attorney) request, the responsibility for these records may be turned over to the client (or attorney) rather than be destroyed.

6.6 The laboratory will accept outside work only when there is no known conflict of interest. Conflicts of interest may include providing results to a competitor of one of Weyerhaeuser's operating units or providing results to an indirect competitor using technology, which is proprietary to Weyerhaeuser.

Those submitting outside work will be informed of the WATS confidentiality policy. Confidential communications/agreements with outside clients will be retained with the service request.

Relevant SOPs, Test Methods, and Documents:
OQ ETHICS
OQ DOCTRL
Outsourced Services-Computer Support

7.0 **Customer Assessment and Communications Processes** (ISO 5.2, 7.2.1, 8.2.1)

7.1 Customer Needs Assessment

7.1.1 Customer Needs Assessment must be a continual process to be effective. Information used in this assessment is acquired from customer feedback, product conformance, corrective/preventive actions, complaints, non-conformance memos, and direct interaction with the clients. This information is summarized and presented at the management review meetings.

7.1.2 The Operations Managers are encouraged to take advantage of every encounter with customers to find out what they want and expect. To accomplish this, WATS' Operations Managers do need assessments through periodic customer surveys. They use these surveys to help determine what the customers want and need to provide valuable, high-quality, innovative, and timely services that meet their requirements.

7.1.3 Needs assessment is a continual process to be effective. The Operations Managers, working with the QA/QC team members, maintain close contact with customers to determine which services to change if the product does not meet their needs.

7.2 Customer Communications Process

7.2.1 WATS encourages its customers to interact and strives to improve customer communications by asking for feedback with its product delivery.

7.2.2 The laboratory management (laboratory manager and operation managers) examines customer feedback during its management reviews.

7.2.3 WATS strives to maintain effective customer communications before, during, and after services are performed to allow dialogue regarding our product.

Relevant SOPs, Test Methods, and Documents:
OQ CUST-SAT.

8.0 **Internal Communications Process** (ISO 5.5.3)

8.1 Internal communication is about change--informing people what is happening within the Weyerhaeuser business groups. Department meetings and the corporate intranet are key tools and these work in conjunction with our website to facilitate knowledge management and knowledge sharing. We use regular WATS department meetings, with the Operations Managers also scheduling smaller periodic team meetings to accomplish this.

8.2 Changes that affect the Quality System are also communicated to all affected staff through regularly held staff meetings. These meetings are held at a team or group, unit, or department level--depending upon complexity or needed clarity. The team level is generally the preferred way, since the groups are small and there is greater opportunity for clarification.

8.3 WATS makes every effort to help employees be a viable part of the organization. The Operations Managers continually strive for a coordinated, comprehensive, long-term communication approach. Staff who understand the basic values and purposes of an organization have an opportunity to make informed decisions and can have direct impact on effort and efficiency.

8.4 An internal communication strategy and its implementation provides a number of benefits to WATS that ensures that the customer receives a quality product. Various tools exist, such as e-mail, memos, reports, department meetings and the Analysis & Testing website are utilized to inform the department of status or activities that affect the Quality System.

Relevant SOPs, test methods, and documents:
OQ DOCTRL.

9.0 **Document Control Process** (ISO 4.2.3)

9.1 Document control to manage the distribution, updating, storage, and modification of documents is important in a quality management system. Effective document control results in reduced costs by eliminating wasted effort due to the use of improper documents. It also results in an increase in customer satisfaction from customers getting expected services. All documents that are important to the operational control and to the technical procedures within the laboratory are controlled.

9.2 Test methods and standard operating procedures are core-operating procedures. These controlled documents have a unique identifier, date of creation/modification and date of issue. These documents are stored electronically and maintained in a database. The preparation of these documents is detailed in the SOP: OQ O-PROC-WRT.

9.3 All documents issued to laboratory personnel as part of the quality system, including those documents that have been revised due to an error, process change or other factors, are approved and authorized prior to issue. The approval is done by one of the Quality Assurance/ Quality Control team members and the personnel having responsibility over the area covered by the document.

9.4 WATS' administrative assistants manage the distribution and retrieval of all documents, as necessary, to insure that only approved documents are in place. The integrated document management system Panagon from FileNet is employed for the document control process to ensure that obsolete documentation is not used in the labs.

9.4.1 Invalid or obsolete documents are removed from all points of use to prevent unintended use. Archived or obsolete documents shall be marked as "ARCHIVED" or "OBSOLETE." Revised documents shall be re-issued as soon as practical to replace the controlled copies in the appropriate lab. A "Revision History" is created in a controlled document to track any changes or updates made.

NOTE: Personal notes or aids written on a document that does not change the technical content are not considered an amendment. However, if such notes are needed, they should be incorporated into the procedure.

9.5 Controlled copies are also accessible via the WATS web site. Copies may be printed as needed, but are only valid for the date printed or for the process duration it is used for. This insures that only valid copies are being used.

Relevant SOPs, Test Methods, and Documents:
OQ DOCTRL
OQ O-PROC-WRT

10. 0 **Record Keeping Process** (ISO 4.2.4)

10.1 In WATS, there are two categories of records: those that pertain to the operation of the lab and those that pertain to generating reports for clients. WATS' administrative assistants maintain and store records as documented in OQ DOCTRL, by managerial delegation.

10.2 A record is the history of what has been done. The quality management system requires retaining records of the evidence that the product fulfills requirements, results of supplier evaluations and actions arising from evaluations. Records are kept for future use as a reference in case of questions related to contractual and legal matters, work techniques, verification of work done, and other parts essential to WATS running smoothly. Our **r**ecord keeping and document control policies insure that we produce a quality product that meets the customers' requirements.

10.3 Operation records include such reports as staff reviews (training records, resumes, performance reviews), purchasing records (subcontractors reports, non-conformance product and investigation reports, expense and capital purchases), and laboratory audits (safety inspections reports, internal audits, state audits and certifications). Besides using this information to maintain and improve laboratory operations, these records are also required to use as reference in case of questions related to contractual and legal matters.

10.4 Laboratory records include calibration data, original observations, derived data, staff records, client reports, non-conformance reports, control charts and any other data that would allow sufficient information to establish audit trails; they also enable identification of factors affecting quality and to allow repeatability of analysis to reproduce sample testing results.

Relevant SOPs, Test Methods, and Documents:
Laboratory Policy and Guidelines Manual (LP&G)
OQ DOCTRL
OQ REPORT GEN

11.0 **Planning Process** (ISO 5.4.1)

11.1 The WATS planning process sets the direction for the quality management system. We start this process by using our WATS Quality Improvement Objectives to identify the current goals of our quality improvement system. These goals are established and reviewed during formal management reviews and at periodic managerial meetings. The purpose of a proposed plan is to address a current problem or pursue a development goal. Completion is acknowledged by posting the year's goals and objectives on the website and communicating the information to employees in staff meetings.

11.2 The WATS planning process facilitates reaching the goals for the laboratory, taking into account resources (raw material, money, technology, and staff), services for the customers (analysis and testing of samples and technical support) and customer satisfaction. These goals include safety, meeting turnaround times for analysis, staff Individual Development Plans (IDP), expense and capital requirements, profit, and customer satisfaction.

11.3 Operations Managers meet with the Laboratory Manager periodically to review the Quality Management System to determine the effectiveness of the quality system and make adjustments to the system. Operations Managers, the Laboratory Manager, and the department's Technical Specialists meet at least once a year to review prior goals and objectives, examine their status, and develop new goals and objectives.

11.4 The planning process is achieved by ongoing reviews of resources, safety audits, internal and external lab audits, and customer satisfaction surveys to determine the effectiveness of the quality system and make adjustments to the system. These reviews are in the form of daily interaction with clients and staff, weekly technical specialist meetings, biweekly team meetings, quarterly staff meetings, biyearly IDP reviews and yearly customer surveys.

11.5 Basic Terms
Planning typically includes use of the following basic terms:

Goals
Goals are specific accomplishments that must be accomplished in total, or in some combination, in order to achieve some larger, overall result preferred from the system, for example, the mission of an organization. (Goals are outputs from the system.)

Strategies or Activities
These are the methods or processes required in total, or in some combination, to achieve the goals. (Strategies are processes in the system.)

Objectives
Objectives are specific accomplishments that must be accomplished in total, or in some combination, to achieve the goals in the plan. Objectives are usually "milestones" along the way when implementing the strategies.

Tasks
Particularly in small organizations, people are assigned various tasks required to implement the plan. If the scope of the plan is very small, tasks and activities are often essentially the same.

Resources (and Budgets)
Resources include the people, materials, technologies, money, etc., required to implement the strategies or processes. The costs of these resources are often depicted in the form of a budget. (Resources are input to the system.)

Relevant SOPs, Test Methods, and Documents:
OP MGT REVIEW

12.0 **Training Process** (ISO 6.2)

12.1 Training records document that laboratory personnel have the proper knowledge and skills to provide ethical quality results safely, and to also be able to function effectively as Weyerhaeuser employees. WATS' training procedure is documented in OQ TRAIN. Certifications and training documents are kept on file for 10 years or term of employment, whichever is longer.

12.2 Operations Managers ensure that technical laboratory staff members are proficient in the activities for which they are responsible. WATS is committed to an active program of on-the-job training, attending job-related courses and seminars, and in continuing education. Membership in professional societies is encouraged. The Weyerhaeuser ROOTS web site ("My Guide") provides additional educational tools under the "Strategic Education" website found under the "My Communities" portal.

12.3 WATS' staff is trained on ethical and legal responsibilities before beginning employment, as documented in OQ ETHICS. This training can be found on "MyGuide" under the Ethics and Business Conduct web page found on "my Roots." Periodically, WATS' staff also completes Weyerhaeuser-provided training in ergonomics and safety and a WATS-offered ethics training.

Relevant documents:
OQ ETHICS
OQ TRAIN
Laboratory Policies and Guidelines (LP&G)
Strategic Education
http://myroots.weyer.com/portal/server.pt?space=CommunityPage&cached=true&parentname=MyPage&parentid=0&in_hi_userid=221637&control=SetCommunity&CommunityID=777&PageID=0
Ethics and Business Conduct
http://www.weyer.com/conduct/training.asp

13.0 **Internal Audit Process** (ISO 8.2.2)

13.1 Internal audits verify that WATS' operations follow the department's quality system and any certification requirements required by external agencies.

13.2 Internal audits are arranged by one or more members of the Quality Assurance/Quality Control team and are conducted by trained and qualified auditors as documented in OQ AUDIT.

13.3 Internal audits evaluate how well the quality standard is applied and the effectiveness of the quality procedures, policies, plans, and instructions.

13.4 Internal audits are scheduled annually, at least one month before the management review. The results of internal audits are presented to management during this review to determine if the department's quality system is suitable and adequate or if changes to the quality system are needed.

Relevant SOPs, Test Methods, and Documents:
OQ AUDIT

14.0 **Management Review Process** (ISO 5.4.1, 5.6)

14.1 The management review process ensures that the quality system is suitable, adequate, and effective and conforms to the regulatory or certification body requirements.

14.2 Management reviews are arranged and conducted by the Quality Assurance/Quality Control team as documented in OQ MGT REVIEW and is conducted at least annually.

14.3 The information presented during the review is used to identify opportunities for improvement within the QMS and assess any need to change the QMS, quality policy, or quality objectives.

14.4 The result from this review process include developing quality improvement objectives, assigning action items to address any deficiencies found during the audits performed, determining resource needs, offering recommendations for the improvement of WATS' products and services, and improving the effectiveness of the quality management system and its processes.

Relevant SOPs, Test Methods, and Documents:
OQ MGT REVIEW
OQ AUDIT

15.0 **Monitoring and Measuring Process** (ISO 7.6)

15.1 All monitoring, analytical and physical testing equipment, whose accuracy affects test data, must be identified (unique name), calibrated, evaluated (verify calibration) and maintained (with maintenance logs). It is the responsibility of the analyst/operator to insure that equipment meets this QA/QC guideline as documented in the Laboratory Policies and Guidelines Manual, notifying technical specialists and operations managers when there is a problem.

15.2 Administrative personnel maintain records of equipment requiring yearly calibration, and remind WATS' team members of calibration schedules. The traceability of measurements contributes to the accuracy, reproducibility, and reliability of testing and of the calibrations performed in the laboratory. This traceability includes a documented SOP for calibration and documented calibration and maintenance in logs and reports. For chemical standards, documented traceability either to national standards or cross checking with different suppliers of standards and documentation of the source of these standards (with lot numbers if applicable) is acceptable.

15.3 If measurements taken with equipment that, at a later date (after reporting), is found to be inaccurate, both the data and equipment must be re-assessed, documented and corrected. All original documentation along with corrected documentation must be retained with the original service request.

Relevant SOPs, Test Methods, and Documents:
Laboratory Policies and Guidelines Manual (LP&G)
OQ O-STDREFMAT

16.0 **Nonconformance and Corrective Action Process** (ISO 8.3, 8.5.2, 8.5.3)

16.1 WATS' nonconformance management process entails documenting supplies, procedures, and analytical and testing results that do not conform to specifications. Two documents address this process: OQ CORRACT for supplies, purchasing and procedures, and OQ NONCONFORM for service results.

16.2 Any team member within WATS may initiate the procedure for corrective actions reports by completing nonconformance forms. These reports are periodically downloaded from the A&T Quality Assurance mailbox into the file management system. This information is used to track, monitor, and troubleshoot the QMS. Management reviews of this data provide a means of improving the quality system by eliminating (or at least minimizing) the occurrence of nonconformities.

Relevant SOPs, Test Methods, and Documents:
OQ CORRACT
OQ NONCONFORM

17.0 **Continual Improvement Process** (ISO 8.4, 8.5)

17.1 The Continual Improvement Process is used by WATS to achieve ever-increasing levels of operational performance and customer satisfaction. Our goal is continual quality improvement.

17.2 Continual improvement is a process of on-going changes that add value to our organization and can also lead to improved customer satisfaction. Corrective action, preventive action, and nonconformance processes can be used to identify possible improvements to methods, application techniques, acceptance criteria, equipment, material, technology, and personnel.

17.3 The continual improvement process encourages all WATS' employees to watch for opportunities to propose improvements and to follow up and make sure that the improvements are implemented.

17.4 The operations managers report to top management on the performance of the quality management system and any need for improvement during regularly scheduled management meetings held throughout the year

17.5 WATS' Quality Improvement Objectives are established and reviewed at the periodic managerial meetings and the formal management reviews. Operations managers work with the laboratory manager to insure that funds and resources are provided to enact changes. In addition, yearly internal audits allow management to identify and introduce any necessary changes or improvements to the organization's effectiveness and efficiency.

Improvement actions can also be characterized as remedial, corrective, preventive, or innovative in nature. The nature of the action(s) is determined by either the operations managers or QA/QC team:

- Corrective Action: An action taken to eliminate the cause(s) of a problem, nonconformity or undesirable situation (reactive). An action taken to prevent recurrence. It can also be an action taken to alleviate or contain a problem, nonconformity or undesirable situation (reactive). Examples are reworking, re-grading, sorting, repairing, rejecting, replacing, and discounting.
- Preventive Action: An action taken to eliminate the cause(s) of a potential problem, potential nonconformity or potential undesirable situation (proactive). Action taken to prevent occurrence.
- Innovative Action: An action taken to change a process or system to better meet objectives (such as improved profitability, lower cost, higher customer satisfaction ratings). Innovative actions may require modifying processes, products or services or the introduction of new processes, products or services (proactive).

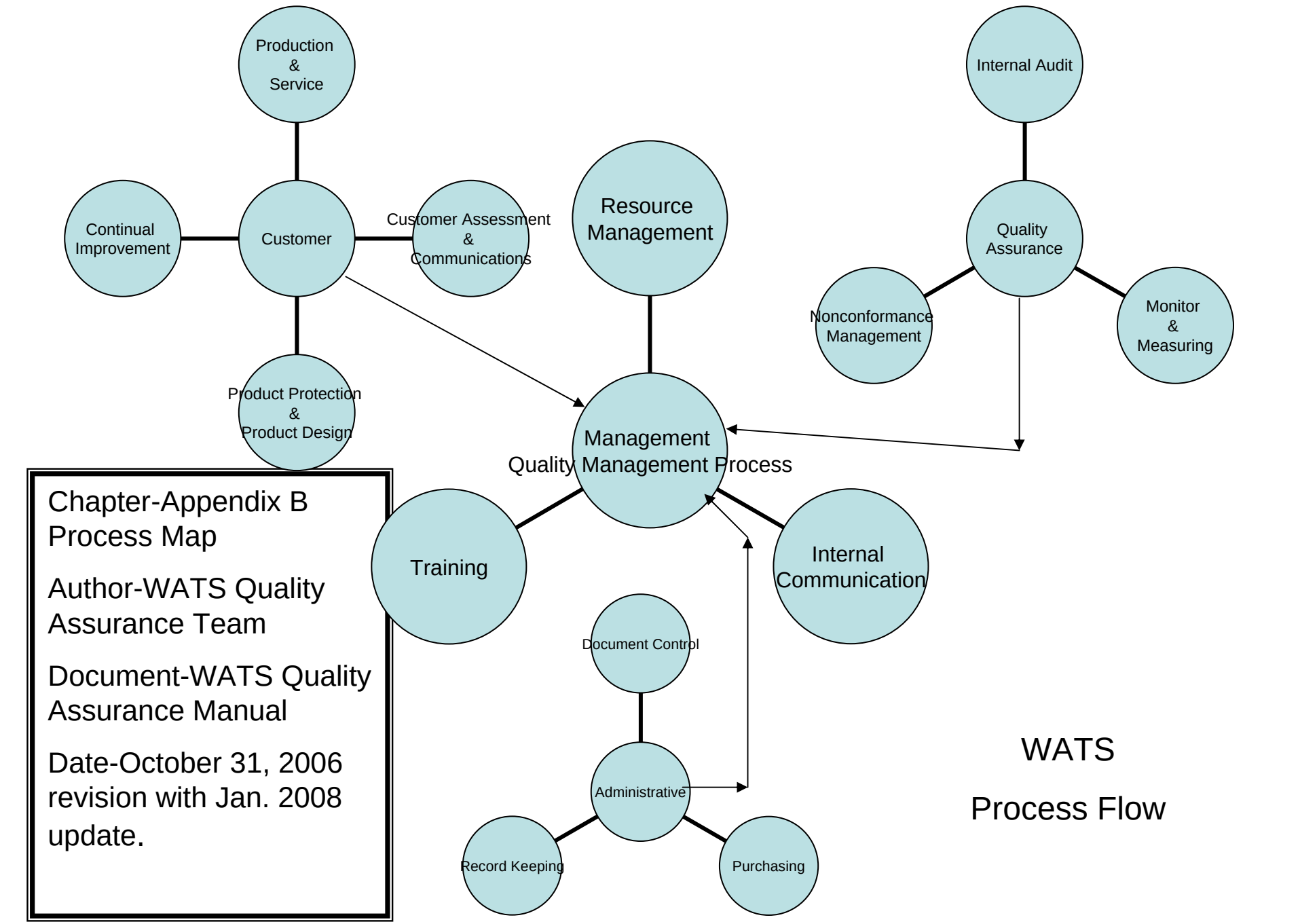
Relevant SOPs, Test Methods, and Documents:
OQ MGT REVIEW

ACRONYMS

ADMIN (2.5.7): Administrative Personnel
APG (2.5.8): Analytical Products Group, Inc. (APG) is an international provider of proficiency testing and quality assurance services.
ASR (6.5): Archived Service Requests
CAR (5.4): Corrective Action Report, A CAR is issued to the responsible follow-up party when an audit non-compliance is found.
CP (1.0)**:** Cellulose Properties, part of Cellulose Fibers Technology Properties (formerly Pulp and Paper Testing, PPT)
CR&D (2.0) Corporate Research and Development
DQO (2.5.3): Data Quality Objectives
ESR (2.5.2): Employee Staffing Requests
IDP (2.0, 11.2): individual development plan is a written action plan designed to help achieve mastery in current assignments and build the capability to assume new, more challenging assignments. The plan targets specific development areas that mutually benefit the employee and Weyerhaeuser.
ISO (2.5.8): International Standards Organization, responsible for the ISO 9000 and other international quality standards.
IT (6.4): Information Technology
LIMS (2.5.3, 2.5.7, 6.3): Laboratory Information Management System
LP&G (5.7.2): Laboratory Policy and Guidelines Manual
myGuide (2.2, 12.2): A Weyerhaeuser HR program. *myGuide* provides a way for employees and managers to find payroll, benefits, and HR-related information that applies specifically to them.
NELAP (2.5.8): National Environmental Laboratory Accreditation Program
OM (2.0): Operations Manager
PACE (4.0): Process to Achieve Capital Excellence. PACE includes the policies, work processes, procedures, and tools used to choose, define, and execute capital projects.
PARS (2.5.1, 2.5.2): Performance Appraisal Reviews
PC/ PC's (6.4): Personal Computer(s)
PM (2.0): Project Manager
PPT (1.0): Pulp and Paper Testing (Cellulose Properties part of Cellulose Fibers Technology)
QA (1.7): Quality Assurance
QA/QC (15.0): Quality Assurance /Quality Control
QC (1.7): Quality Control
QMS (FORWARD, 14.3, 16.2): Quality Management System
RADAR (2.0): A disciplined and safe process for responding to upset conditions (i.e., unplanned, non-routine work)

SMO (2.5.3): Sample Management Office
SOP (1.3, 2.5.8, 5.3) Standard Operating Procedure
SQL (5.7): A standard computer language for accessing and manipulating databases
SR (2.5.5): Service Request (Work Order)
TM (2.5.8): Test Method
TS (2.5.4): Technical Specialist
UM (2.0): Unit Manager
WATS (Introduction, 5.7.2): Weyerhaeuser Analysis and Testing Services

Activities (11.5): See Strategies
Cellulose Properties (1.0): Formerly called Pulp and Paper Testing, PPT. Part of Cellulose Fibers Technology.
Record (10.2): A record is a written history of the work we have performed. Some examples of records include such reports as staff reviews (training records, resumes, performance reviews), purchasing records (subcontractors reports, non-conformance product and investigation reports, expense and capital purchases), and laboratory audits (safety inspections reports, internal audits, state audits and certifications).
Goals (11.0): Goals are specific accomplishments that must be accomplished in total, or in some combination, in order to achieve some larger, overall result preferred from the system, for example, the mission of an organization. (Goals are outputs from the system.) These set the direction for the quality management system.
Product (3.0): WATS' product is defined as test data in the form of reports, which are delivered to our clients.
Strategies (11.5): Are the methods or processes required in total, or in some combination, to achieve the goals. (Strategies are processes in the system.)
Objectives (11.5): Objectives are specific accomplishments that must be accomplished in total, or in some combination, to achieve the goals in the plan. Objectives are usually "milestones" along the way when implementing the strategies.
Tasks (11.5): Particularly in small organizations, people are assigned various tasks required to implement the plan. If the scope of the plan is very small, tasks and activities are often essentially the same.
Resources (11.5): Resources include the people, materials, technologies, money, etc., required to implement the strategies or processes. (Resources are input to the system.)
Budgets (11.5): See Resources
Roots (2.2, 12.2): Weyerhaeuser website


Production & Service
Continual Improvement
Customer
Customer Assessment & Communications
Resource Management
Internal Audit
Quality Assurance
Nonconformance Management
Monitor & Measuring
Product Protection & Product Design
Management Quality Management Process
Training
Internal Communication
Document Control
Administrative
Record Keeping
Purchasing
Chapter-Appendix B Process Map
Author-WATS Quality Assurance Team
Document-WATS Quality Assurance Manual
Date-October 31, 2006 revision with Jan. 2008 update.
WATS
Process Flow

Index

360°	2.5.2
administrative	Quality Policy Statement, 4.0, 6.0, 9.4, 10.1, 15.2
Analysts	2.5.6
annual(ly)	2.4, 12.3, 13.4, 14.2
audits	14.4
audits, internal	2.5.8, 5.7, 10.3, 13.0, 17.5
audits, lab	10.3, 11.4
audits, state	10.3
audits, safety	11.4
continual improvement	Forward, 3.3, 17.0
calibration	6.3, 10.4, 15.1, 15.2
quality goals	1.8
confidential(ity)	5.5, 6.1, 6.6
confidential policy	6.6
department meetings	8.0
IDP	2.0, 11.0
ISO	Quality Policy Statement, Forward, 1.0, 2.5.8, 5.7.2
Laboratory Policies and Guidelines	5.7.2, 15.1
Laboratory Information Management System	6.3 (see LIMS)
Laboratory Manager	1.6, 2.4, 2.5.2, 2.5.8, 5.2.1, 7.2.2, 11.3, 12.5,
LIMS	2.5.3, 2.5.4, 2.5.6, 2.5.7, 6.3, 6.4,
LP&G	(see Laboratory Policies and Guidelines)
maintenance logs	15.0
Management Representative	2.5.1, 2.5.8, 5.2.2
mentor support	13
Operations Manager	2.5.1, 2.5.2, 2.5.3, 2.5.8, 5.2.2, 5.2.3, 7.1.2, 7.1.3, 8.1, 8.3,11.3, 12.215.1, 17.4, 17.5 (see Laboratory Manager)
People Lead	2.5.1 (see Managemenet Representative)
Project Manager	2.5.2, 2.5.3, 2.5.4, 2.5.5, 2.5.6, 2.5.7, 2.5.8, 3.2, 5.2.2, 5.2.3
planning process	11.1, 11.2
purchasing	Forward, 4.0, 10.3, 16.1
quality improvement objectives	11.1, 14.4, 17.5
QMS	Quality Policy Statement, Forward, 14.3, 16.2 (see quality management system)

quality management system	Quality Policy Statement, Forward, 1.3, 2.1, 2.5.8, 9.1, 10.2, 11.1, 11.3, 14.4, 17.4,
Sample Management	2.5.5 (see SMO)
SMO	2.5.3, 2.5.5, 2.5.7
SOP	1.3, 2.5.4, 2.5.8, 5.3, 15.2
standard operating procedure	(see SOP)
subcontractors	10.3
Technicians	2.5.6
Technical Specialist	2.5.4
time off	13
training documents	6.5, 12.1
training procedure	12.1
training	Quality Policy Statement, 2.5.2, 6.2, 12.1
training, on-the-job	12.2
training, records	10.3, 12.1
training, resources	2.4
training, technical	2.5.4
WATS operations	13.1
WATS Process Flowchart	3.2, appendix
WATS Quality Assurance Manual	Forward
WATS Quality System	2.1
website	9.5
work-related event	13

QUALITY ASSURANCE MANUAL

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Effective Date: March 1, 2008

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Initials: _____ Date: _____

2.0 TABLE OF CONTENTS

Section	Heading	Page
1.0	Title Page with Provision Signatures	1
2.0	Table of Contents	2
3.0	Introduction and Company Quality Assurance Policy	4
4.0	Program Description	5
5.0	Professional Conduct and Ethical Practices	10
6.0	Project Organization and Responsibilities	11
7.0	Information Management	14
8.0	Sample Management	16
9.0	Analytical Procedures	30
10.0	Calibration Procedures and Frequency	34
11.0	Quality Control	39
12.0	Data Reduction, Validation, and Reporting	46
13.0	Performance and System Audits	54
14.0	Preventive Maintenance	58
15.0	Corrective Action	60
16.0	Quality Assurance Reports	62
17.0	Personnel Training	63
18.0	References for Analytical Procedures	67

Tables		Page
Table 6-1	Summary of Technical Experience and Qualifications	13
Table 8-1	Sample Preservation and Holding Times	20
Table 12-1	Descriptions of CAS Data Deliverables	53
Table 13-1	Current CAS Performance and System Audit Programs	56

Figures Page

Figure 4-1 CAS/Kelso Laboratory Floor Plan 9
Figure 8-1 Chain of Custody Form 27
Figure 8-2 Cooler Receipt and Preservation Check Form 28
Figure 8-3 Tier V Project Specification Form 29
Figure 15-1 Corrective Action Report 61
Figure 17-1 Initial Demonstration of Proficiency Requirements 66

Appendices Page

Appendix A List of QA Program Documents A1
Appendix B Organizational Chart and Resumes of Key Personnel B1
Appendix C Major Analytical Equipment C1
Appendix D Preventive Maintenance Procedures D1
Appendix E SOP List and List of NELAP Accredited Methods E1

3.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is an employee-owned professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, and other material.

It is a policy at CAS that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. This goal is achieved by ensuring that adequate Quality Control (QC) procedures are used throughout the monitoring process, and by establishing a means to assess performance of these Quality Control and other QA activities. Policies and procedures are established in order to meet the quality objectives of clients, accrediting authorities, and certifying organizations. The Quality System is established to meet the requirements of The NELAC Institute (TNI) National Environmental Laboratory Accreditation Program (NELAP).

CAS maintains control of analytical results by adhering to written standard operating procedures (SOPs) and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory.

CAS is a network of laboratories. In addition to the Kelso, WA facility, to which this manual is applicable, CAS also operates laboratories in California, Florida, New York, Arizona, and Texas.

The information in this document has been organized according to the format described in *EPA Requirements for Quality Management Plans, EPA QA/R-2,* USEPA, 2001; and *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5,* USEPA, 2001.

4.0 PROGRAM DESCRIPTION

The purpose of the QA program at CAS is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement of CAS:

> "The mission of Columbia Analytical Services, Inc., is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development and company commitment."

In support of this mission, our QA program addresses all aspects of laboratory operations, including laboratory organization and personnel, standard operating procedures, sample management, sample and quality control data, calibration practices, standards traceability data, equipment maintenance records, method proficiency data (such as method detection limit studies and control charts), document control/storage and staff training records.

4.1 Facilities and Equipment

CAS features over 45,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each work space. Also, CAS minimizes laboratory contamination sources by employing janitorial and maintenance staff to ensure that good housekeeping and facilities maintenance are performed. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Shipping and Receiving/Purchasing
- Sample Management Office, including controlled-access sample storage areas
- Inorganic/Metals Sample Preparation Laboratories (2)
- Inorganic/Metals “clean room” sample preparation laboratory
- ICP-AES Laboratory
- ICP-MS Laboratory
- AA Laboratory
- Water Chemistry & General Chemistry Laboratories (3)
- Semi-volatile Organics Sample Preparation Laboratory
- Gas Chromatography/High Performance Liquid Chromatography Laboratories (2)

- Gas Chromatography/Mass Spectrometry Laboratory
- Petroleum Hydrocarbon Laboratory
- Semi-volatile Organics Drinking Water Laboratories (2)
- Volatile Organics Laboratory
  - Separate sample preparation laboratory
  - Access by semi-volatile sample preparation staff only after removing lab coat and solvent-contaminated gloves, etc.
- Microbiology Laboratory
- Laboratory Deionized Water Systems (2)
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas
- Information Technology (IT) and LIMS

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 4-1 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix C lists the major equipment, illustrating the laboratory's overall capabilities and depth.

4.2 Technical Elements of the Quality Assurance Program

The Quality Assurance Program provides a platform on which technical operations are based. The program provides laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained. This includes method approvals and audit administration. In addition, internal audits are performed to assess compliance with policies and procedures. Standard Operating Procedures (SOPs) are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix A.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, and preventative maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas. The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

4.3 Operational Assessments

There are a number of methods used to assess the laboratory and its daily operations. In addition to the routine quality control (QC) measurements to measure quality, the senior laboratory management examines a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients. On-time performance, report quality, training, and Quality Assurance are a few of the items that are used to assess performance from an external perspective. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

CAS utilizes a number of different methods to ensure that adequate resources are available in anticipation of the demand for service. Regularly scheduled senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating resources to achieve the required results. All Requests for Proposal (RFP) documents are reviewed by the Project Chemist and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Plans (QAPPs) if available. A weekly status meeting is also conducted with the laboratory staff by the Client Services Manager to inform the staff of the status of incoming work, future projects, or project requirements.

4.4 Document Control

Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL).* The procedures described in the SOP include distribution, tracking, filing, and copyrighting of CAS controlled documents. The requirements of the SOP apply to all standards preparation logbooks, instrument maintenance logbooks, run logbooks, standard operating procedures (SOPs), quality assurance manuals (QAMs), quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled CAS documents.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the Quality Assurance Manager, or designee, and ensure that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file.

CAS maintains a records system that ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data Archiving (ADM-ARCH).*

4.5 Subcontracting

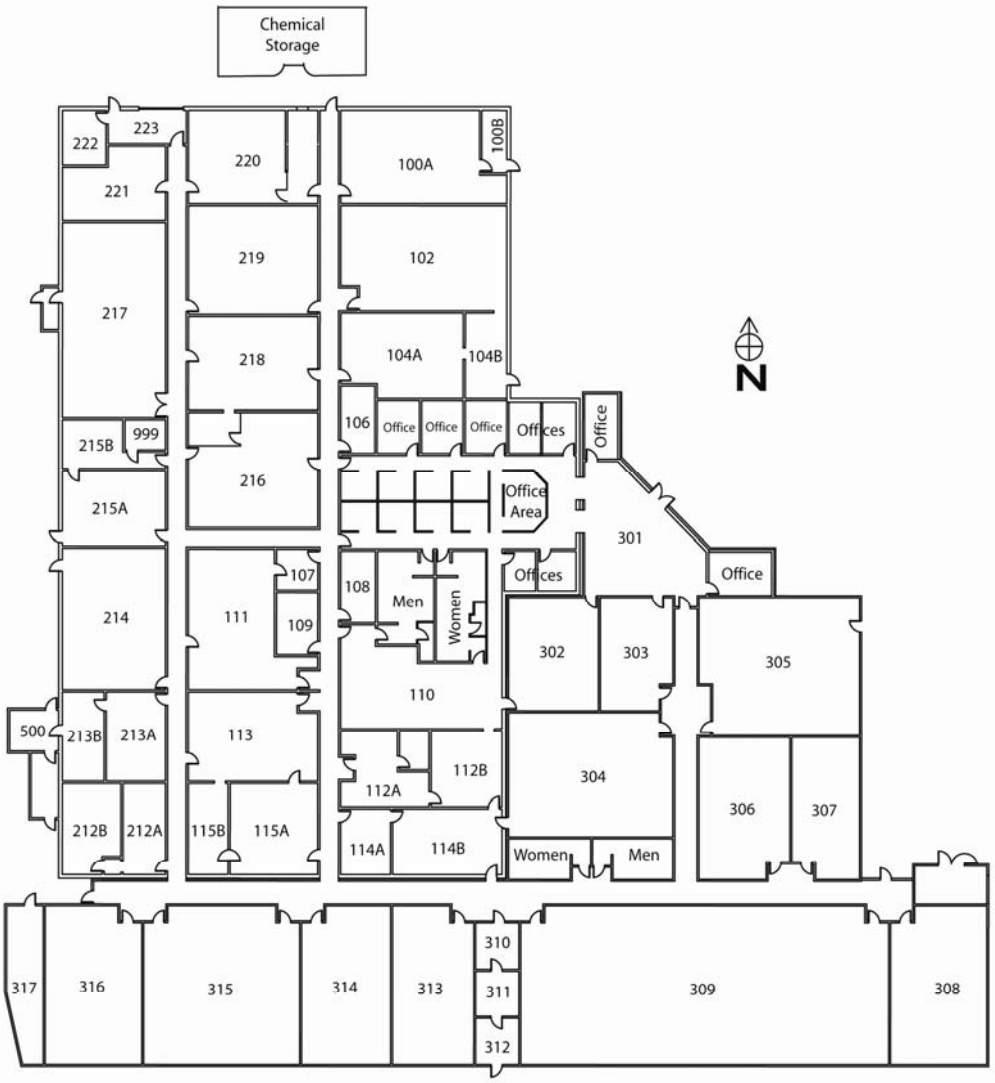
Analytical services are subcontracted when CAS/Kelso needs to balance workload or when the requested analyses are not performed by CAS/Kelso. Subcontracting is only done with the knowledge and approval of the client. Subcontracting to another CAS laboratory is preferred over external-laboratory subcontracting. Further, sub-contracting is done using capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories Outside of CAS Network (ADM-SUBLAB).* The Corporate Quality Assurance staff is responsible for qualifying and oversight of subcontract laboratories.

4.6 Procurement

The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned (by the laboratory user) as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. The procedures for purchasing and procurement are described in the *SOP for Purchasing through CAS Purchasing Department in Kelso (SOP ADM-PUR).* Also, refer to section 10.4 for a discussion of reference materials.

Figure 4-1
CAS/Kelso Laboratory Floor Plan

Room #	
100A	Semivolatile GC Lab
100B	Electrical Room
102	Semivolatile GC Lab
104A	Petroleum Hydrocarbon Lab
104B	Petroleum Hydrocarbon Lab
106	Deionized Water System Room
107	Storage
108	Zero Headspace Extraction
109	Storage
110	Lunch Room
111	General Chemistry Lab
112A	Microbiology Lab
112B	Copy Center
113	Metals Lab
114A	Sample Preparation
114B	Sample Preparation
115A	Metals Lab
115B	Metals Lab
212A	Metals Lab
212B	Low Level Mercury Lab
213A	Sample Preparation
213B	Sample Preparation
500	Janitorial Room
214	General Chemistry Lab
215A	Drinking Water Lab
215B	Data Storage
999	Computer Room
216	Data Review Area
217	Sample Storage
218	Volatiles Lab
219	Semivolatile GC/MS Lab
220	Semivolatile GC/MS Lab
221	Drinking Water Lab
222	Purchasing
223	General Receiving
301	Lobby
302	Lunch Room
303	Conference Room
304	Sample Storage
305	Sample Receiving
306	Pharmaceutical A
307	Pharmaceutical B
308	Pharmaceutical C
309	Organics Extractions Lab
310	Communication Room
311	Electrical Room
312	Fire Room/Water System
313	Glass Room
314	Clean Room
315	Metals Digestion Room
316	Tissue Preparation
317	Mechanical



5.0 PROFESSIONAL CONDUCT AND ETHICAL PRACTICES

One of the most important aspects of the success of CAS is the emphasis placed on the integrity of the data provided and services performed. To promote product quality, employees are required to comply with certain standards of conduct and ethical practices. The following examples of CAS policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action. Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.

- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible. Employee discipline is progressive in its severity and each situation is handled individually in that the discipline is designed to fit the circumstances. Potential disciplinary actions may include a verbal warning, written warning, a second written notice (more severe and more strongly worded than a warning), suspension without pay, demotion, or termination.

- It is the responsibility of all CAS employees to safeguard sensitive company and client information. The nature of our business and the well being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action.

All employees are required to sign and adhere to the requirements set forth in the CAS *Confidentiality and Conflicts of Interest Employee Agreement* and the CAS *Commitment to Excellence in Data Quality Policy.* All employees receive in-house ethics training and are periodically reminded of their data quality and ethical conduct responsibilities.

CAS makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the CAS Employee Handbook. This includes the CAS Ombudsman Program, the CAS Open Door Policy, and the use of flexible work hours. Operational assessments are regularly made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads (Section 4.3). Procedures for subcontracting work are established, and within the CAS laboratory network additional capacity is typically available for subcontracting, if necessary.

6.0 ORGANIZATION AND RESPONSIBILITIES

The CAS/Kelso staff, consisting of approximately 110 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that the laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks.

CAS is committed to providing an environment that encourages excellence. Everyone within CAS shares responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 6-1 lists the CAS/Kelso personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of these key personnel, can be found in Appendix B.

- The role of the Laboratory Director is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the Kelso facility. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.
- The responsibility of the Quality Assurance Manager (QAM) is to oversee implementation of the quality program and to coordinate QA activities within the laboratory. The QAM works with laboratory production units to establish effective quality control and assessment plans. The QAM has the authority to stop work in response to quality problems. The QAM is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and coordinating the annual review of each SOP; maintaining QA records such as metrological records, archived logbooks, PT sample results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; performing internal QA audits; preparing QA activity reports; etc. The QAM reports directly to the Laboratory Director. The QAM also interacts with the CAS Quality Assurance Director. It is important to note that when evaluating data, the QAM does so in an objective manner and free of outside, or managerial, influence.

  The Chief Quality Officer (CQO) is responsible for the overall QA program at all the CAS laboratories. The CQO is responsible for ensuring that annual internal audits are performed at each CAS laboratory; maintaining a data base of information about state certifications and accreditation programs; writing laboratory-wide SOPs; maintaining a data base of CAS-approved subcontract laboratories; providing assistance to the laboratory QA staff and laboratory managers; preparing a quarterly QA activity report; etc.

- In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are the Client Services Manager or Organics Department Manager (for the Laboratory Director) and the CQO or Laboratory Director (for the QA Manager).

- The Environmental Health and Safety Officer (EH&S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S officer is also designated as the Chemical Hygiene Officer. The EH&S Officer has a dotted-line reporting responsibility to CAS' EH&S Director.

- The Client Services and Sample Management Office Manager is responsible for the Client Services Department (customer services/project chemists, and Electronic Data Deliverables group) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specification to final deliverables. The sample management office handles all the activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.

- The Project Chemist is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project chemist is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the CAS laboratory and administrative staff to ensure that client-specific needs are understood, and that the services CAS provides are properly executed and satisfy the requirements of the client.

- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a quality control program based upon the unique requirements within the department. Each Department Manager and Supervisor has the responsibility to ensure that quality control functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each department manager has the authority to stop work in response to quality problems in their area. Analysts have the responsibility to carry out testing according to prescribed methods, SOPs, and quality control guidelines particular to the laboratory in which he/she is working.

- The Sample Management Office plays a key role in the laboratory QA program by maintaining documentation for all samples received by the laboratory, and by assisting in the archival of all laboratory results. The sample management office staff is also responsible for the proper disposal of samples after analysis.

- Information Technology (IT) staff are responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

Table 6-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Jeff Christian, B.S.	29	Laboratory Director
Lee Wolf, B.S.	22	Quality Assurance Manager
Lynda Huckestein, B.S.	19	Client Services Manager Sample Management Office Manager
Jeff Coronado, B.S.	18	Metals Department Manager
Todd Poyfair, B.S.	16	General Chemistry & Extractions Department Manager
Jeff Grindstaff, B.S.	19	Organics Chromatography & Mass Spectrometry Department Manager
Loren Portwood, B.S.	17	Organics Drinking Water Department Manager
Eileen Arnold, B.A.	26	Environmental Health and Safety Officer
Ed Wilson, B.A.	34	CAS Information Technology Director
Gary Ward, M.S.	32	CAS Chief Quality Officer
Steve Vincent, B.S.	32	CAS President

7.0 INFORMATION MANAGEMENT

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. CAS management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies. Appendix C lists major automated data processing equipment.

7.1 Software Quality Assurance Plan

CAS has defined practices for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the CAS Software Quality Assurance Plan (SQAP). The purpose of the SQAP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. Key components of configuration management plan are policies for controlling the software version that is in use in the laboratory.

7.2 IT Support

The local CAS Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitors the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data. A software inventory is maintained. Additional IT responsibilities are described in the SQAP.

In addition to the local IT department, CAS corporate IT provides support for network-wide systems. CAS also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

7.3 Information Management Systems

CAS has various systems in place to address specific data management needs. The CAS Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system is used to establish and define sample identification, analysis specifications, and provide a means of sample tracking. This system is used during sample login to generate the internal Service Request. The Service

Request provides a summary of client information, sample information, required analyses, work instructions, deliverable requirements and other necessary information provided on the chain of custody. The LIMS also is the basis for valuable sample tracking mechanisms used throughout the laboratory. Laboratory analysts generate responsibility reports from the LIMS and perform internal chain of custody via the LIMS.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2003® domain). This provides a reliable, easily maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. The server is also used for data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats, including Staged Electronic Data Deliverable (SEDD) format.

7.4 Backup and Security

CAS laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving. Differential backups are performed on all file server information once per day, Sunday through Thursday. Full backups are performed each Friday night. Tapes are physically stored in a locked media cabinet within a locked, temperature controlled computer room, with every other full backup also securely stored offsite.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person's areas of responsibility. Passwords are required on all systems. No direct external, non-CAS access is allowed to any of our network systems.

The external e-mail system and Internet access is established via a single gateway to discourage unauthorized entry. CAS uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent. The external messaging system operates through a single secure gateway. Email attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a firewall for added security. Virus screening is performed continuously on all network systems.

8.0 SAMPLE MANAGEMENT

8.1 Sampling and Sample Preservation

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. CAS recommends that clients follow sampling guidelines described in 40 CFR 136, 40 CFR 141, USEPA SW-846, and state-specific sampling guidelines, if applicable. Sampling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

CAS uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IV for hazardous waste samples; USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; EPA 40CFR parts 136 and 141; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples (see Section 18 for complete citations). The container, preservation and holding time information for these references is summarized in Table 8-1 for soil, water, and drinking water. The current EPA CLP Statement of Work should be referred to for CLP procedures. Where allowed by project sampling and analysis protocols (such as Puget Sound Protocols) the holding time for sediment, soil, and tissue samples may be extended for a defined period when stored frozen at -20°C.

CAS routinely provides sample containers with appropriate preservatives for our clients. Containers are purchased as precleaned to a level 1 status, and conform to the requirements for samples established by the USEPA. Certificates of analysis for the sample containers are available to clients if requested. Reagent water used for sampling blanks (trip blanks, etc.) and chemical preservation reagents are tested by the laboratory to ensure that they are free of interferences and documented. Our sample kits typically consist of foam-lined, precleaned shipping coolers, (cleaned inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, (VOC vials are placed in a specially made, foam holder), chain-of-custody (COC) forms, and custody seals. Container labels and custody seals are provided for each container.

Figure 8-1 shows the chain-of-custody form routinely used at CAS and included with sample kits. For large sample container shipments, the containers may be shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to CAS. The proper preservative is added to the sample containers prior to shipment, unless otherwise instructed by the client.

If any returning shipping cooler exhibits an odor or other abnormality after receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Containers exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. CAS keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. CAS also provides courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When CAS ships environmental samples to other laboratories for analysis each sample bottle is wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during shipping. The sample management office (SMO) follows formalized procedures for maintaining the chain of custody of the sample(s) (*SOP for Chain of Custody for Sample Transfer between Laboratories* [SOP ADM-COC]), proper packaging and shipment, specification of proper methodology, etc. Blue or gel ice is the only temperature preservative used by CAS, unless otherwise specified by the client or receiving laboratory.

8.2 Sample Receipt and Handling

Standard Operating Procedures are established for the receiving of samples into the laboratory. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received. Complete documentation of all sample storage is maintained in order to preserve the integrity of the samples.

Once samples are delivered to the CAS sample management office (SMO), a Cooler Receipt and Preservation Check Form (CRF - See Figure 8-2 for an example) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.);
- Sample containers checked for integrity (broken, leaking, etc.);

- Sample is clearly marked and dated (bottle labels complete with required information);
- Appropriate containers (size, type) are received for the requested analyses;
- The minimum amount of sample material is provided for the analysis.
- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (Assessment of proper preservation of VOC containers is performed by lab personnel).

Samples are logged into a Laboratory Information Management System (LIMS). Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and COC documents. Potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When the Project Chemist and client have reached a satisfactory resolution, the login process may continue and analysis may begin. During the login process, each sample is given a unique laboratory code and a service request form is generated. The LIMS generates a Service Request that contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. The service request is reviewed by the appropriate Project Chemist for accuracy, completeness, and consistency of requested analyses and for client project objectives.

Samples are stored as per method requirements until they undergo analysis, unless otherwise specified, using various refrigerators or freezers, or designated secure areas. CAS has five walk-in cold storage units which house the majority of sample containers received at the laboratory. In addition, there are four additional refrigerators, including dedicated refrigerated storage of VOC samples. The dedicated storage areas for VOC samples are monitored using storage blanks, as described in the *SOP for VOA Storage Blanks (VOC-BLAN)*. CAS also has six sub-zero freezers capable of storing samples at -20° C primarily used for tissue and sediment samples requiring specialized storage conditions. The temperature of each sample storage unit is monitored daily and the data recorded in a bound logbook. Continuous-graph temperature recorders have also been placed in the walk-in refrigerators to provide a permanent record of the storage conditions to which samples are exposed.

CAS adheres to the method-prescribed or project-specified holding times for all analyses. The sampling date and time are entered into the LIMS system at the time of sample receipt and login. Analysts then monitor holding times by obtaining analysis-specific reports from the LIMS. These reports provide holding time information on all samples for the analysis, calculated from the sampling date and the holding time requirement. To document holding time compliance, the date and time analyzed is printed or written on the analytical raw data. For analyses with a holding time prescribed in hours it is essential that the sample collection time is provided, so holding time compliance can be demonstrated. If not, the sample collection time is assumed as the earliest in the day (i.e. the most conservative).

Unless other arrangements have been made in advance, upon completion of all analyses and submittal of the final report, aqueous samples and sample extracts are retained at ambient temperature for 30 days, soil samples are retained at ambient temperature for 60 days, and tissue samples are retained frozen for 3 months. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures outlined in the *CAS Environmental Health and Safety Manual.* All waste produced at the laboratory, including the laboratory's own various hazardous waste streams, is treated in accordance with applicable local and Federal laws. Documentation is maintained for each sample from initial receipt through final disposal to ensure that an accurate history of the sample from "cradle to grave" is available.

8.3 Sample Custody

Sample custody transfer at the time of sample receipt is documented using chain-of-custody (COC) forms accompanying the samples. During sample receipt, it is also noted if custody seals were present. This is described in the *SOP for Sample Receiving (SMO-GEN)*. Figure 8-1 is a copy of the chain-of-custody form routinely used at CAS.

Facility security and access is important in maintaining the integrity of samples received at CAS/Kelso. Access to the laboratory facility is limited by use of locked exterior doors with a coded entry, except for the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area with locked doors with a coded entry. The CAS facility is equipped with an alarm system and CAS employs a private security firm to provide nighttime and weekend security.

A barcoding system is used to document internal sample custody. Each person removing or returning samples from/to sample storage while performing analysis is required to document this custody transfer. The system uniquely identifies the sample container and provides an electronic record of the custody of each sample. For sample extracts and digestates the analyst documents custody of the sample extract or digestate by signing on the benchsheet, or custody record, that they have accepted custody. The procedures are described in the *SOP for Sample Tracking and Internal Chain of Custody (SMO-SCOC).*

8.4 Project Setup

The analytical method(s) to be used for sample analysis are chosen based on the client's requirements. Unless specified otherwise, the most recent versions of reference methods are used. For SW-846 methods, some projects may require the most recent *promulgated* version, and some projects may require the most recent *published* version. The Project Chemist will ensure that the correct method version is used. LIMS codes are chosen to identify the analysis method used for analysis. The Project Chemist ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the LIMS generated Service Request. To communicate and specify project-specific requirements, a Tier V form (Figure 8-3) is used and accompanies the service request form.

Table 8-1
Sample Preservation and Holding Times

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Colilert (Standard Methods)	W, DW	P, Bottle or Bag	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Coliform, Fecal and Total (Standard Methods)	W, DW	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Fecal Streptococci (SM 9230B)	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Inorganic Tests				
Acidity (SM 2310B)	W	P,G	Cool, 4°C	14 days[EPA]
Alkalinity (SM 2320B)	W, DW	P,G	Cool, 4°C	14 days[EPA]
Ammonia (SM 4500NH3)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand (SM 5210B)	W	P,G	Cool, 4°C	48 hours
Bromate (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	28 days
Bromide (EPA 300.1)	W, DW	P,G	None Required	28 days
Chemical Oxygen Demand (SM 5220C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride (EPA 300.0)	W, DW	P,G	None Required	28 days
Chloride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Chlorine, Total Residual (SM 4500Cl F)	W, DW	P,G	None Required	24 hours
Chlorite (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	14 days
Chlorophyll-A (SM 11200H)	W	G Amber	Cool, 4°C	Analyze immediately
Chromium VI (EPA 7196A)	W	P,G	Cool, 4°C	24 hours
Color (SM 2120B)	W, DW	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination (EPA 335.4, 9010, 9012) (SM 4500CN E,G)	W, DW	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable (SM 4500CN I)	W	P,G	Cool, 4°C, NaOH to pH >12	14 days
Ferrous Iron (CAS SOP)	W, DW	G Amber	Cool, 4°C	24 hours
Fluoride (EPA 300.0)	W, DW	P,G	None Required	28 days
Fluoride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Hardness (SM 2340C)	W, DW	P,G	HNO_3 to pH<2	6 months
Hydrogen Ion (pH) (SM 4500H B)	W, DW	P,G	None Required	Analyze immediately
Kjeldahl and Organic Nitrogen (ASTM D3590-89)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Nitrate (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrate (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Nitrate-Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrite (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrite (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Orthophosphate (EPA 365.3)	W, DW	P,G	Cool, 4°C	Analyze immediately
Oxygen, Dissolved (Probe) (SM 4500O G)	W, DW	G, Bottle and Top	None Required	Analyze immediately
Oxygen, Dissolved (Winkler)	W, DW	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Perchlorate (EPA 314.0)	W, DW	P,G	Protect from temp. extremes	28 days
Phenolics, Total (EPA 420.1)	W	G Only	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorus, Total (EPA 365.3)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Residue, Total (EPA 160.3 & SM 2540B)	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS) (SM 2540C)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS) (SM 2540D)	W	P,G	Cool, 4°C	7 days
Residue, Settleable (SM 2540F)	W	P,G	Cool, 4°C	48 hours
Residue, Volatile (EPA 160.4)	W	P,G	Cool, 4°C	7 days
Silica (SM 4500SiO2 C)	W	P Only	Cool, 4°C	28 days
Specific Conductance (EPA 120.1 & SM 2510B)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 300.0)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Sulfide (SM 4500S2 F)	W	P,G	Cool, 4°C, Add Zinc Acetate plus Sodium Hydroxide to pH>9	7 days
Sulfite (SM 4500SO3 B)	W	P,G	None Required	24 hours
Surfactants (MBAS) (SM 5540C)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin (SM 5550B)	W	P,G	Cool, 4°C	28 days
Turbidity (EPA 180.1)	W, DW	P,G	Cool, 4°C	48 hours

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Metals				
Metals, except CrVI and Mercury	W, DW	P,G	HNO_3 to pH<2	6 months
(EPA 200.7, 200.8, 200.9, 6010, 6020)	S	G, Teflon-Lined Cap	Cool, 4°C	6 months
Chromium VI (EPA 7195/7191)	W	P,G	Cool, 4°C	24 hours
Mercury	W	P,G	HNO_3 to pH<2	28 days
(EPA 245.1, 7470, 7471, 1631E)	S	P,G	Cool, 4°C	28 days
Organic Tests				
Oil and Grease, Hexane Extractable Material (EPA 1664)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Carbon, Total (EPA 415.1, 9060 & SM 5310C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Halogens, Total (EPA 9020)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (EPA 1650B)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO_3 to pH<2	6 months
Petroleum Hydrocarbons, Total (EPA 8015)	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H_2SO_4 to pH<2	7 days until extraction; 40 days after extraction
	S	G, Teflon-Lined Cap	Cool, 4°C	14 days until extraction; 40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Petroleum Hydrocarbons, Volatile (Gasoline-Range Organics) (EPA 8015)	W	G, Teflon-Lined Septum Cap	Cool, 4°C, HCl to pH<2 No Headspace	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C Minimize Headspace	14 days
Purgeable Halocarbons (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	No Residual Chlorine Present: HCl to pH<2, Cool, 4°C, No Headspace Residual Chlorine Present: 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE) (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	No Residual Chlorine Present: HCl to pH<2, Cool, 4°C, No Headspace Residual Chlorine Present: 10% $Na_2S_2O_3$, HCl to pH<2, Cool 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Acrolein, Acrylonitrile, Acetonitrile (EPA 624, 8260)	W	G, Teflon-Lined Septum Cap	Adjust pH to 4-5, Cool, 4°C, No Headspace	14 days
EDB and DBCP (EPA 8260)	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics) (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Alcohols and Glycols (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Acid Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Base/Neutral Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Polynuclear Aromatic Hydrocarbons (EPA 625, 8270, 8310)	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Organochlorine Pesticides and PCBs (EPA 608, 8081)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Organophosphorus Pesticides (EPA 8141)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides (EPA 8141)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Herbicides (EPA 8151)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Organotins (CAS SOP)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Chlorinated Phenolics (EPA 1653A)	W	G, Teflon-Lined Cap	H_2SO_4 to pH<2, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Resin and Fatty Acids (NCASI 85.02)	W	G, Teflon-Lined Cap	NaOH to pH $\geq$10, Cool, 4°C[g]	30 days until extraction; 30 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Drinking Water Organics				
Purgeable Organics (EPA 524.2)	DW	G, Teflon-Lined Septum Cap	Ascorbic Acid, HCl to pH≤2, Cool, 4°C, No Headspace	14 days
EDB, DBCP, and TCP (EPA 504.1)	DW	G, Teflon-Lined Septum Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	14 days
Carbamates, Carbamoyloximes (EPA 531.1)	DW	G, Amber, Teflon-Lined Cap	1.8 mL monochloroacetic acid to pH<3; 80 mg/L $Na_2S_2O_3$ if Res.Cl.; Cool, 4°C	28 days
Chlorinated Herbicides (EPA 515.4)	DW	G, Amber, Teflon-Lined Cap	If Res.Cl, 2mg/4omL NaS; Cool, <6°C	14 days until extraction; 21 days after extraction
Chlorinated Pesticides (EPA 508.1, 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Diquat and Paraquat (EPA 549.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$ if Res.Cl., Cool, 4°C,	7days until extraction; 21 days after extraction
Endothall (EPA 548.1)	DW	G, Amber, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; 14 days after extraction
Glyphosate (EPA 547)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$, Cool, 4°C	14 days
Haloacetic Acids (EPA 552.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L NH_4Cl, Cool, 4°C	14 days until extraction; 7 days after extraction
Semivolatile Organics (EPA 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Toxicity Characteristic Leaching Procedure (TCLP)				
Mercury (EPA 1311/7470)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	28 days until extraction; 28 days after extraction
Metals, except Mercury (EPA 1311/6010)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	180 days until extraction; 180 days after extraction
Volatile Organics (EPA 1311/8260)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C Minimize Headspace TCLP extract: Cool, 4°C, HCl to pH<2, No Headspace	14 days until extraction; 14 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Toxicity Characteristic Leaching Procedure (TCLP)				
Semivolatile Organics (EPA 1311/8270)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark[g] TCLP extract: Cool, 4°C, Store in Dark[g]	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Organochlorine Pesticides (EPA 1311/8081)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Chlorinated Herbicides (EPA 1311/8151)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction

a For EPA SW-846 methods the method number is listed generically, without specific revision suffixes.
b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste
c P = Polyethylene; G = Glass
d For chlorinated water samples
e The maximum holding time is dependent upon the geographical proximity of sample source to the laboratory.
f Fourteen days until extraction for soil, sediment, and sludge samples.
g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate.

Figure 8-1
Chain of Custody Form

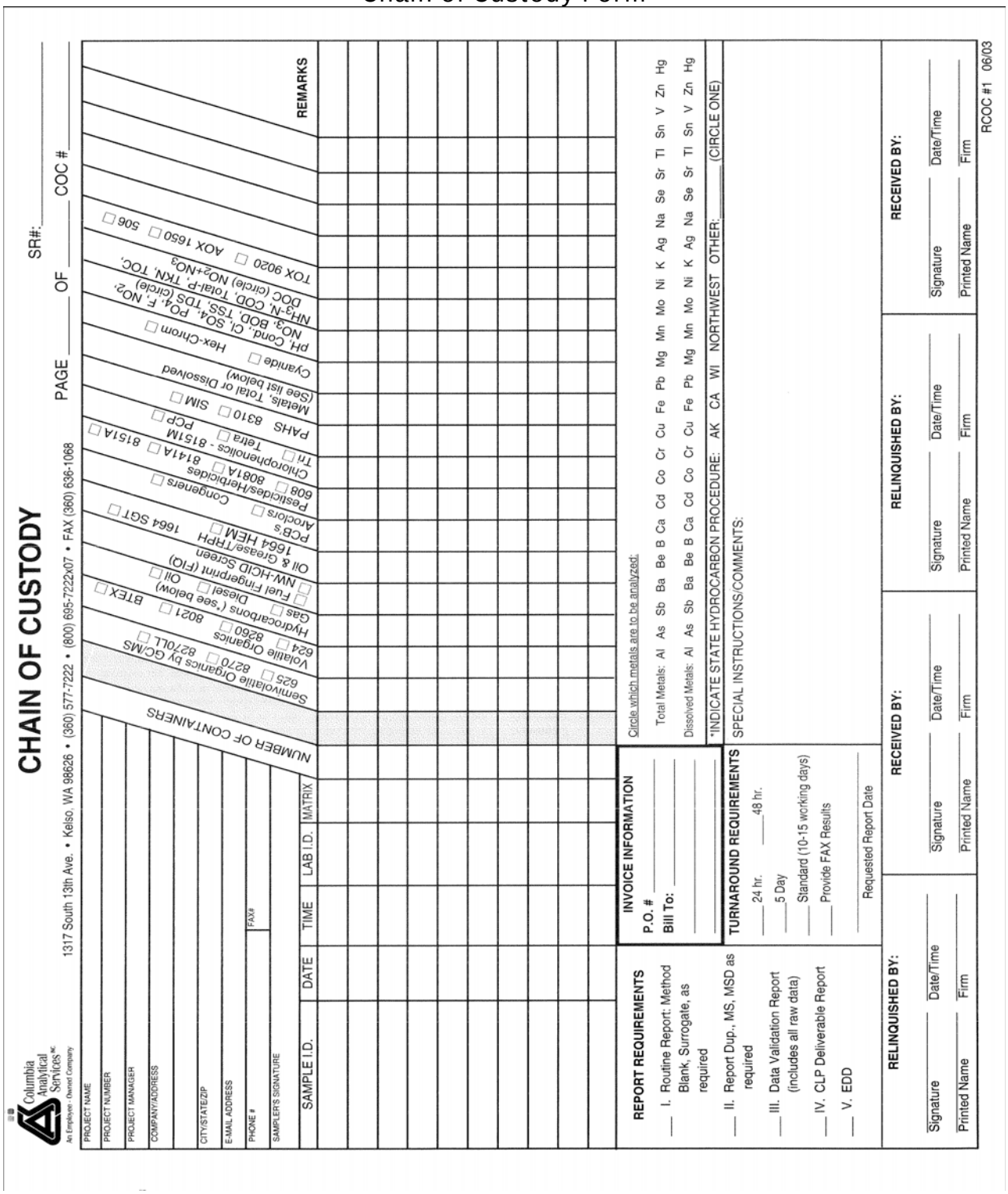


Figure 8-2

Columbia Analytical Services, Inc. PC_____
Cooler Receipt and Preservation Form

Client / Project:_____ Service Request ***K08***_____

Received:_____ Opened:_____ By:_____

1. Samples were received via? ***US Mail*** ***Fed Ex*** ***UPS*** ***DHL*** ***GH*** ***GS*** ***PDX*** ***Courier*** ***Hand Delivered***
2. Samples were received in: (circle) ***Cooler*** ***Box*** ***Envelope*** ***Other***_____ ***NA***
3. Were custody seals on coolers? NA Y N If yes, how many and where?_____
   If present, were custody seals intact? Y N If present, were they signed and dated? Y N
4. Is shipper's air-bill filed? If not, record air-bill number:_____ NA Y N

_____ _____ _____

5. **Temperature of cooler(s) upon receipt (°C):** _____ _____ _____ _____
   Temperature Blank (°C): _____ _____ _____ _____
6. If applicable, list Chain of Custody Numbers: _____ _____ _____ _____
7. Were custody papers properly filled out (ink, signed, etc.)? NA Y N
8. Packing material used. ***Inserts*** ***Bubble Wrap*** ***Gel Packs*** ***Wet Ice*** ***Sleeves*** ***Other***_____
9. **Did all bottles arrive in good condition (unbroken)?** *Indicate in the table below.* NA Y N
10. Were all sample labels complete (i.e analysis, preservation, etc.)? Y N
11. Did all sample labels and tags agree with custody papers? *Indicate in the table below* Y N
12. **Were the correct types of bottles used for the tests indicated?** NA Y N
13. Were all of the preserved bottles received at the lab with the appropriate pH? *Indicate in the table below* NA Y N
14. Were VOA vials and 1631 Mercury bottles checked for absence of air bubbles? *Indicate in the table below.* NA Y N
15. **Are CWA Microbiology samples received with >1/2 the 24hr. hold time remaining from collection?** NA Y N
16. Was C12/Res negative? NA Y N

Sample ID on Bottle	Sample ID on COC	Sample ID on Bottle	Sample ID on COC

Sample ID	Bottle Count	Bottle Type	Out of Temp	Head-space	Broken	pH	Reagent	Volume added	Reagent Lot Number	Initials

Additional Notes, Discrepancies, & Resolutions:_____

_____

_____

Figure 8-3
Tier V Form

Client :

Project Chemist :

Project Name :

Service Request :

Project Number :

SMO LimsTemplate ID :

Project Description :

QAPP/SOW Information :

Reporting

Tier Level :

PDF:

Report to :

In result field use :

EDD :

Flagging Requirements :

Other Requirements :

Sample Considerations

Sample Limitations :

Sample Prep/Analysis :

Non-Standard Holdtimes :

Historical Data :

Comments :

9.0 ANALYTICAL PROCEDURES

CAS employs methods and analytical procedures from a variety of sources. The primary method references are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB, and online updates for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 18.0. Other published procedures, such as state-specific methods, program-specific methods (such as Puget Sound Protocols), or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by CAS is described in SOPs specific to each method. A list of SOPs and NELAP-accredited methods are given in Appendix E. Further details are described below.

9.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

CAS maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements. Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA Manager maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents. The procedures for document control are described in the *SOP for Document Control* (ADM-DOC_CTRL). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in the *SOP for Making Entries into Logbooks and onto Benchsheets* (ADM-DATANTRY). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

9.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the project chemist handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The project chemist is responsible for documenting the approved or allowed deviation from the SOP by placing a detailed description of the deviation attached to the quotation or in the project file and also providing an appropriate comment on the service request when the samples are received.

For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the laboratory director, or other level of authority. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

9.3 Modified Procedures

CAS strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

9.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that CAS has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples, are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.

2) All (field) samples in a batch are of the same matrix.

3) The QC samples to be processed with the (field) samples include:

   a) Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination.

   b) Laboratory Control Sample (a.k.a. Laboratory Fortified Blank)

      Function: Assessment of method performance

   c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*

      Function: Assessment of matrix bias

   d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

      Function: Assessment of batch precision

   * A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.

4) A single lot of reagents is used to process the batch of samples.

5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.

6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours.

7) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.

8) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).

9) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.

10) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.

11) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

9.5 Specialized Procedures

CAS not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are trace-level Mercury and methylmercury analyses, reductive precipitation metals analysis, specialized GC/MS analyses, LC/MS analyses, and ultra-low level organics analyses (including PAHs, pesticides and PCBs).

9.6 Sample Cleanup

CAS commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods for cleanup of sample extracts for organics analysis are routinely used to minimize or eliminate interferences that may adversely affect sample results and data usability.

10.0 CALIBRATION PROCEDURES AND FREQUENCY

All equipment and instruments used at CAS are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files.
Records are maintained to provide traceability of reference materials.

Equipment which has been subjected to overloading or mishandling, or has been shown by verification or otherwise to be defective; is taken out of service until it has been repaired. The equipment is placed back in service only after verifying by calibration that the equipment performs satisfactorily. An evaluation of the effect of this defect on previous calibrations or tests is made and documented appropriately.

10.1 Temperature Control Devices

Temperatures are monitored and recorded for all of the temperature-regulating support equipment such as sample refrigerators, freezers, and standards refrigerators. Bound record books are kept which contain daily-recorded temperatures, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the *SOP for Support Equipment Monitoring and Calibration (SOP ADM-SEMC).* The SOP also includes the use of acceptance criteria and correction factors.

Where the operating temperature is specified as a test condition (such as ovens, incubators, evaporators) the temperature is recorded on the raw data. All thermometers are identified according to serial number, and the calibration of these thermometers is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a professional metrology organization on an annual basis.

10.2 Analytical Balances

The calibration of each analytical balance is checked by the user each day of use with three Class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high levels bracketing the working range. Records are kept which contain the recorded measurements, identification of the balance, acceptance criteria, and the initials of user who performed the check. The procedure for performing these measurements and use of acceptance criteria is described in the SOP ADM-SEMC. The weights are recertified using NIST traceable standards by a professional metrology organization on an annual basis.

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Analytical balances are serviced on a semi-annual basis by a professional metrology organization. New certificates of calibration for each balance are issued to the laboratory on a semi-annual basis.

10.3 Water Purification Systems

CAS uses two independent water purification systems is designed to produce deionized water meeting method specifications. One system consists of a series of pumps, filters, and resin beds designed to yield deionized water meeting the specifications of ASTM Type II water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. A second system consists of pumps, filters, and treatment components designed to yield deionized water meeting the specifications of ASTM Type I water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Following a written SOP, the status of each system is monitored continuously for conductivity and resistivity with an on-line meter and indicator light, and readings recorded daily in a bound record book. The meter accuracy is verified annually. Deionizers are rotated and replaced on a regular schedule. Microbiology water is checked at a point downstream of the purification system at a tap in the laboratory, and monitoring documented.

10.4 Source and Preparation of Standard Reference Materials

All analytical measurements generated at CAS are performed using materials and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. All sampling containers provided to the client by the laboratory are purchased as precleaned (Level 1) containers, with certificates of analysis available for each bottle type. This information is provided to the client when requested.

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A_2LA. CAS relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, etc. are examples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination. The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP No. ADM-DATANTRY). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material (see section 11.3.5).

10.5 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards. Analyses of calibration standards, initial and continuing calibration verification standards, and inter-element interference check samples are carried out as specified in the applicable method SOP and analytical method (i.e. EPA 200.7, 6010B, 6010C, CLP SOW, etc.).

10.6 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Each element of interest is calibrated for using a blank and a single standard. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard (CCV) is analyzed with every ten samples.

10.7 Atomic Absorption Spectrophotometers (AAS)

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples. Initial calibration points cannot be “dropped” from the resulting calibration curve.

10.8 GC/MS Systems

All GC/MS instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method. Compounds selected as system performance check compounds (SPCCs) must show a method-specified response factor in order for the calibration to be considered valid. Calibration check compounds (CCCs) must also meet method specifications for percent difference from the multipoint calibration. For isotope dilution procedures, the internal standard response(s) and labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked using bromofluorobenzene (BFB) for volatile organic chemical (VOC) analysis, or decafluorotriphenylphosphine (DFTPP) for semi-volatile analysis. Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.9 Gas Chromatographs and High Performance Liquid Chromatographs

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. All GC and HPLC instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise). The lowest standard is equivalent to the method reporting limit; additional standards define the working range of the GC or LC detector. Results are used to establish response factors (or calibration curves) and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples, unless otherwise specified by the reference method. *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.10 LC/MS Systems

Calibration and tuning procedures are included in analytical SOPs written specifically for these tests. In general, multiple concentration levels for the analytes of interest are used to generate calibration curves. All reference materials used for this function are vendor-certified standards. Calibration and tuning verification is performed at SOP-defined intervals. Any other system performance checks are described in the applicable SOP. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.11 UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5-point calibration curve including a blank. Initial calibration points cannot be “dropped” from the resulting calibration curve. Correlation coefficients must meet method or SOP specifications before analysis can proceed. Independent calibration verification standards (ICVs) are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods at CAS include total phenolics, phosphates, surfactants and tannin-lignin.

10.12 Flow Injection Analyzer (automated colorimetric analysis)

A minimum of six standards and a blank are used to calibrate the instrument for cyanide analysis. A blank and (minimum of) five standards are used to calibrate the instrument for all other automated chemistries. Initial calibration points cannot be “dropped” from the resulting calibration curve. Standard CAS acceptance limits are used to evaluate the calibration curve prior to sample analysis.

10.13 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a calibration curve with the method-specified number of points (or more). Initial calibration points cannot be “dropped” from the resulting calibration curve. A correlation coefficient of $\geq$ 0.995 for the curve is required before analysis can proceed. Quality Control (QC) samples that are routinely analyzed include blanks and laboratory control samples. The target analytes typically determined by the IC include nitrate, nitrite, chloride, fluoride, sulfate and drinking water

inorganic disinfection byproducts. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

10.14 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, Analytical Products Group® QC samples (or equivalent) and duplicates.

10.15 Ion-selective electrode

The method-prescribed numbers of standards are used to calibrate the electrodes before analysis. The slope of the curve must be within acceptance limits before analysis can proceed. Quality Control samples that are routinely analyzed include blanks, LCSs and duplicates.

10.16 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP for Checking Pipet Calibration*. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks.

10.17 Other Instruments

Calibration for the total organic carbon (TOC), total organic halogen (TOX), and other instruments is performed following manufacturer's recommendations and applicable SOPs.

11.0 QUALITY CONTROL

A primary focus of Columbia Analytical Services Quality Assurance (QA) Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis of field samples, acceptable method performance is established by performing demonstration of capability analyses and performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. CAS has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the methodology or are statistically derived based on the laboratory's actual historical data obtained from the various QC measurements for each analytical method. The Quality Control objectives are defined below.

11.1 Quality Control Objectives

11.1.2 Demonstration of Capability - Where required by mandatory test method, regulation, or accreditation protocols, a demonstration of capability (DOC) is made prior to using any test method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control reference material or quality control sample is obtained. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results or MDL study results may be used to meet this requirement, provided acceptance criteria is met.

11.1.3 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or

caused by an artifact of the measurement system (e.g., contamination). CAS utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement

11.1.4 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

11.1.5 Control Limits - The control limits for accuracy and precision originate from two different sources: For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are updated periodically when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the Quality Assurance Manager. The new control limits replace the previous limits and data is assessed using the new values. The current acceptance limits for accuracy and precision are available from the laboratory and on the accompanying CD-ROM. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

11.1.6 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. CAS has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample. These include the *SOP for Subsampling and Compositing of Samples* and the *SOP for Tissue Sample Preparation*. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

11.1.7 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using CAS or project-specified data qualifiers.

11.2 Method Detection Limits and Method Reporting Limits

Method Detection Limits (MDL) for methods performed at CAS/Kelso are determined annually, and may change slightly from year to year. The MDLs are determined by following the *SOP for the Determination of Method Detection Limits and Limits of Detection,* which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using MDL verification samples. The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. the lower limit of quantitation). Therefore, analyses are calibrated to the MRL, or lower. To take into account day-to-day fluctuations in instrument sensitivity, analyst performance, and other factors, the MRL is established at three times the MDL (or greater). The current MDLs and MRLs are available from the laboratory.

11.3 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below. In addition, a number of other quality control processes that may impact analytical results are also described below.

11.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < ½ MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

11.3.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

11.3.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of either analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

11.3.4 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

11.3.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration with newly prepared standard(s) but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used in the calibration. Once it is determined that there is no reference material defect or systematic error in preparation of the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). ICVs are also analyzed in accordance with method-specific requirements.

11.3.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

11.3.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

11.3.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.9 Laboratory Control Samples (a.k.a. Laboratory Fortified Blanks)

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free solid (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples. The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.10 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is(are) added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$Recovery\ (\%) = (S - A) \times 100 \div T$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

11.3.11 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$Relative\ Percent\ Difference\ (RPD) = (S1 - S2) \times 100 \div S_{ave}$$

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

11.3.12 Interference Check Samples

An interference check sample (ICS) is a solution containing both interfering and analyte elements of known concentration that can be analyzed to verify background and

interelement correction factors in metals analyses. The ICS is prepared to contain known concentrations (method or program specific) of elements that will provide an adequate test of the correction factors. The ICS is analyzed at the beginning and end of an analytical run or at a method-specified frequency. Results must meet method criteria and any project-specific criteria.

11.3.13 Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the method reporting limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

11.3.14 Control Charting

The generation of control charts is routinely performed at CAS. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Charting Quality Control Data* (ADM-CHRT).

11.3.15 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at CAS undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at CAS; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

CAS reports the analytical data produced in its laboratories to the client via the certified analytical report (CAR). This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

12.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the hardcopy is forwarded to the supervisor or second qualified analyst, who reviews the data for errors. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable, a final copy of the report is printed and signed by the laboratory supervisor, departmental manager or designated laboratory staff. The entire data package is then placed into the appropriate service request file, and an electronic copy of the final data package is forwarded to the appropriate personnel for archival. Data review procedures are described in the *SOP for Laboratory Data Review Process.*

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP ADM-DATANTRY).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the "before" and "after" integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration of Chromatographic Peaks* (SOP ADM-INT).

12.2 Confirmation Analysis

12.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This is limited to petroleum hydrocarbon analyses (e.g., gasoline and diesel) and does not include polychlorinated biphenyls.

- The sample meets all of the following requirements:

    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring. Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.

    2. All analytes have been previously analyzed in sample(s) from the same source (within the last year), identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out. The documents indicating previous confirmation must be available for review.

12.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods

    1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).

    2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.

- GC/MS Methods - Two criteria are used to verify identification:

    1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.

    2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

12.3 Data Review and Validation

The integrity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, AFCEE QAPP protocols, and project-specific QAPPs.

- Method Calibration – Following the analysis of calibration blanks and standards according to the applicable SOP the calibration correlation coefficient, average response factor, etc. is calculated and compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.

- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications, or a new initial calibration is performed. For DoD projects, the concentration of these two consecutive must be varied as required by the DoD QSM, Version 3.

- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.

- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) it is verified that the result is within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. For sample and sample duplicate analyzed for precision, the calculated RPD is compared to the specified limits. The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. For metals, additional measures described in the applicable SOP may be taken to further evaluate results (dilution tests and/or post-digestion spikes). Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are performed the MRL is elevated accordingly and qualified. The MRL must meet project requirements.

- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. It is verified that the result is within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are performed the MRL is elevated accordingly and qualified. The MRL must meet project requirements.

- Surrogate Results (Organic) – Following sample analysis and calculations the percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable and other sample evaluation is complete, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present. If no matrix interferences are present and there is no cause for the outlier, the sample is reprepared and reanalyzed. However, if the recovery is above the upper control limit with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.

- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used. Despite the use of homogenizing procedures prior to sample preparation or analysis, the sample may not be homogenous or duplicate sample containers may not have been sample consistently. If non-homogenous, the result is reported with a qualifier about the homogeneity of the sample. Also, the results are compared to the MRL. If the results are less than five times the MRL, the results are reported with a qualifier that the high RPD is due to the results being near the MRL. If the sample is homogenous and results above five times the MRL, the samples and duplicates are reanalyzed. If re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.

- Laboratory Control Sample Results – Following analysis of the LCS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. The source of the problem is identified and, depending on the source of the problem, the LCS and the associated batch is reanalyzed or re-prepared and reanalyzed.

- Matrix Spike Results – Following analysis of the MS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

12.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package is reviewed by a trained chemist. Prior to release of the report to the client, the project chemist reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw data, along with a copy of the final report, is filed in project files by service request number for archiving. CAS maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data are calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The *SOP for Data Reporting and Report Generation* addresses the flagging and qualification of data. The CAS-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the project chemist to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Chemist verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Chemist accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the CAS client.

12.5 Documentation

CAS maintains a records system which ensures that all laboratory records of analysis data retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. The archiving system is described in the *SOP for Data Archiving*.

12.5.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes the following items for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Logbook ID number for the appropriate standards;
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

12.6 Deliverables

In order to meet individual project needs, CAS provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 12-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) to following specialized deliverables:

- ADEC – Alaska Department of Conservation specified data package
- ACOE/HTRW – Army Corps of Engineers specified data package and reporting requirements (HTRW, CERP, FUDS, etc.)
- AFCEE – Air Force Center for Environmental Excellence project-specific reporting

When requested, CAS provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. CAS is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the hard-copy report for accuracy.

Table 12-1
Descriptions of CAS Standard Data Deliverables

Tier I. Routine Certified Analytical Report (CAR) includes the following:

1. Transmittal letter
2. Sample analytical results
3. Method blank results
4. Surrogate recovery results and acceptance criteria for applicable organic methods
5. Chain of custody documents
6. Dates of sample preparation and analysis for all tests

Tier II and IIA. In addition to the Tier I Deliverables, this CAR includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Tier IIA also includes Laboratory Control Sample (LCS) result(s) with calculated recovery and including associated acceptance criteria

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:

1. Case narrative
2. Calibration records and results of initial and continuing calibration verification standards, with calculated recoveries
3. Results of laboratory control sample (LCS) or Quality Control check sample, with calculated recovery and/or associated acceptance limit criteria
4. Results of calibration blanks or solvent blanks (as appropriate to method)
5. Summary forms for associated QC and calibration parameters
6. Copies of all raw data, including extraction/preparation bench sheets, chromatograms, and instrument printouts. For GC/MS, this includes tuning criteria and mass spectra of all positive hits. Results and spectra of TIC compounds will be included upon request.

Tier IV. CLP-Level Data Validation Package.

A complete Data Validation Package containing all sample results, quality control and calibration results, and raw data necessary to fulfill all deliverable requirements of an EPA Contract Laboratory Program (CLP) data package.

13.0 PERFORMANCE AND SYSTEM AUDITS

Quality audits are an essential part of CAS/Kelso's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

13.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of CAS/Kelso are conducted regularly by various regulatory agencies and clients. Table 13-1 summarizes some of the major programs in which CAS/Kelso participates. Programs and certifications are added as required. Additionally, internal system audits of CAS/Kelso are conducted regularly under the direction of the Quality Assurance Manager. The internal audit procedures are described in the *SOP for Internal Audits*. The internal audits are performed as follows:

- Comprehensive lab-wide system audit – performed annually. This audit is conducted such that systems, technical operations, hardcopy data, and electronic data are assessed.
- Hardcopy report audits – minimum of 3 per quarter.
- Electronic audit trail reviews – each applicable instrument per quarter.

All audit findings, and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Electronic data audits may be performed in conjunction with hardcopy data audits. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices and files, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

13.2 Performance Audits

CAS/Kelso also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. CAS routinely participates in the following studies:

- Water Pollution (WP) and additional water parameters, 2 per year.
- Water Supply (WS) PT studies, 2 per year.
- Hazardous Waste/Soil PT studies, 2 per year.
- Underground Storage Tank PT studies, 2 per year.
- Microbiology (WS and WP) PT studies, 2 per year.
- Other studies as required for specific certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are reviewed by the Quality Assurance Manager, Laboratory Director, the laboratory staff, and the CAS Quality Assurance Director. For any results outside acceptance criteria, the analysis data is reviewed to identify a possible cause for the deficiency, and corrective action is taken and documented.

Table 13-1
Current CAS Performance and System Audit Programs

Federal and National Programs

- The TNI (The NELAC Institute) National Environmental Laboratory Accreditation Program (NELAP) Accredited Drinking Water, Non-Potable Water, Solid & Hazardous Waste, and Biological Tissue Laboratory
- Naval Facilities Engineering Service Center
  Validated Laboratory for NFESC Parameters
- U.S. Air Force, Air Force Center for Environmental Excellence (AFCEE)
  Approved Laboratory for AFCEE Projects
- U.S. Army Corps of Engineers
  Approved Laboratory for USACE Projects
- U.S. EPA Region 8
  Approved Drinking Water Laboratory

State and Local Programs

- State of Alaska, Department of Environmental Conservation
  UST Laboratory, Lab I.D. UST040
- State of Arizona, Department of Health Services
  License No. AZ0339
- State of Arkansas, Department of Environmental Quality
  Certified Environmental Laboratory, Lab I.D. 88-0637
- State of California, Department of Health Services, Environmental Laboratory Accreditation Program
  Certification No. 2286
- State of Colorado, Department of Public Health and Environment
  Certified Drinking Water Laboratory
- State of Florida, Department of Health
  Primary NELAP Accreditation No. E87412
- State of Georgia, Department of Natural Resources
  Certified Drinking Water Laboratory
- State of Hawaii, Department of Health
  Certified Drinking Water Laboratory
- State of Idaho, Department of Health and Welfare
  Certified Drinking Water Laboratory
- State of Indiana, Department of Health
  Certified Drinking Water Laboratory, Lab I.D. C-WA-01
- State of Louisiana, Department of Environmental Quality
  Accredited Environmental Laboratory, Lab I.D. 3016
- State of Louisiana, Department of Health and Hospitals
  Accredited Drinking Water Laboratory, Lab I.D. LA080001
- State of Maine, Department of Human Services
  Certified Environmental Laboratory, Lab I.D. WA0035
- State of Michigan, Department of Environmental Quality
  Certified Drinking Water Laboratory, Lab I.D. 9949

Table 13-1 (continued)

State and Local Programs (continued)

- State of Minnesota, Department of Health
  Certified Environmental Laboratory, Lab I.D. 053-999-368
- State of Montana, Department of Health and Environmental Sciences
  Certified Drinking Water Laboratory, Lab I.D. 0047
- State of Nevada, Division of Environmental Protection
  Certified Drinking Water Laboratory, Lab I.D. WA35
- State of New Jersey, Department of Environmental Protection
  Accredited Environmental Laboratory, Lab I.D. WA005
- State of New Mexico, Environment Department
  Certified Drinking Water Laboratory
- State of North Carolina, Department of Environment and Natural Resources
  Certified Environmental Laboratory, Lab I.D. 605
- State of Oklahoma, Department of Environmental Quality
  General Water Quality/Sludge Testing, Lab I.D. 9801
- State of Oregon, ORELAP Laboratory Accreditation Program
  Accredited Environmental Laboratory, Lab I.D. WA200001
- State of Pennsylvania Department of Environmental Protection
  Registered Environmental Laboratory
- State of South Carolina, Department of Health and Environmental Control
  Certified Environmental Laboratory, Lab I.D. 61002
- State of Utah, Department of Health, Division of Laboratory Services
  Accredited Environmental Laboratory
- State of Washington, Department of Ecology, Environmental Laboratory Accreditation Program
  Accreditation No. C1203
- State of Wisconsin, Department of Natural Resources
  Accredited Environmental Laboratory, Lab I.D. 998386840

14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at CAS (e.g., ICP/MS and ICP systems, GC/MS systems, atomic absorption spectrometers, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at CAS contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at CAS before it maybe used for sample analysis. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Equipment records also include a copy of the manufacturer's manual(s) and dates and results of calibrations.

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at CAS. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the section supervisor. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each laboratory section maintains a critical parts inventory. The parts inventories include the items needed to perform the preventive maintenance procedures listed in Appendix D.

This inventory or “parts list” also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as gas chromatography/mass spectrometry jet separators and electron multipliers and ICP/MS nebulizer. When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the table in Appendix D for a list of preventive maintenance activities and frequency for each instrument.

15.0 CORRECTIVE ACTION

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). Failure to meet established analytical controls, such as the quality control objectives outlined in Section 11, prompts corrective action. In general, corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the Quality Assurance Manager may examine and pursue alternative solutions. In addition, the appropriate project chemist may be notified in order to ascertain if contact with the client is necessary.

In the event that analyses produce nonconformances with data or results, the problem and the corresponding corrective actions taken are documented on Nonconformity and Corrective Action Reports (See Figure 15-1) following the requirements in the SOP for Corrective Action (SOP No. ADM-CA). This form is utilized to document corrective actions in response to out-of-control situations. The Quality Assurance Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the Quality Assurance Manager. The Quality Assurance Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate project chemist is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

In addition to internal communication of data issues, the laboratory also maintains a system for dealing with customer complaints. The person who initially receives the feedback (typically the project chemist) is responsible for documenting the complaint. If the project chemist is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QA Manager for final resolution. The complaint and resolution are documented. The procedure is described in the *SOP for Handling Customer Feedback* (ADM-FDBK).

Corrective action due to a performance audit or a check sample problem is initiated by the Quality Assurance Manager; the affected laboratory supervisors and managers are promptly informed of performance audit results requiring corrective action.

Figure 15-1

Columbia Analytical Services, Inc.

Nonconformity and Corrective Action Report

NONCONFORMITY

NCAR No. _____

PROCEDURE (SOP or METHOD): EVENT DATE: _____

EVENT: ☐ Missed Holding Time ☐ QC Failure ☐ Lab Error (spilled sample, spiking error, etc.)
☐ Method Blank Contamination ☐ Login Error ☐ Project Management Error
☐ Equipment Failure ☐ Unacceptable PT Sample Result
☐ SOP Deviation ☐ Other (describe):

SAMPLES / PROJECTS / CUSTOMERS / SYSTEMS AFFECTED

DETAILED DESCRIPTION

ORIGINATOR: _____ DATE: _____

PROJECT MANAGER(S): _____ NOTIFIED BY: _____ DATE: _____

CORRECTIVE ACTION AND OUTCOME

Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the particular Nonconformity and prevent its reoccurrence. Include Project Manager instructions here.

Is the data to be flagged in the Analytical Report with an appropriate qualifier? ☐ **No** ☐ **Yes**

APPROVAL AND NOTIFICATION

Supervisor Verification and Approval of Corrective Action _____ Date: _____
Comments:

QA PM Verification and Approval of Corrective Action _____ Date: _____
Comments:

Customer Notified by ☐ Telephone ☐ Fax ☐ E-mail ☐ Narrative ☐ Not notified

Project Manager Verification and Approval of Corrective Action _____ Date: _____
Comments:

(Attach record or cite reference where record is located.)

Original: QA PM
NCAR 2007.doc 9/11/2007

Page 1 of 1

Photocopies: Supervisor and Customer File
File Name: NCAR 2007

16.0 QUALITY ASSURANCE REPORTS

Quality assurance requires an active, ongoing commitment by CAS personnel at all levels of the organization. Communication and feedback mechanisms are designed so that analysts, supervisors and managers are aware of QA issues in the laboratory. Analysts performing routine testing are responsible for generating a data quality report with every analytical batch processed. This report contains explicit documentation of the various controls used during the analysis. This report also allows the analyst to provide appropriate notes and/or a case narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) (see Section 15.0) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and corrected if possible.

It is the responsibility of each laboratory unit to provide the project chemist with a final report of the data, accompanied by signature approval. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate project chemist, who in turn reviews the entire collection of analytical data for completeness. The project chemist must also review the entire body of data to ensure that any and all client-specified objectives were successfully achieved. A case narrative may be written by the project chemist to explain any unusual problems with a specific analysis or sample, etc.

The Quality Assurance Manager (QAM) provides overview support to the project chemists as required (e.g., contractually specified, etc.). The QAM is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities. The QAM provides the Laboratory Director with quarterly reports that summarize the various QA/QC activities that occurred during the previous quarter. The report addresses such topics as the following:

- Status, schedule, and results of internal and external audits;
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;
- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

Any operational or quality assurance problems noted by the Laboratory Director are then addressed during the senior staff operations meetings with all appropriate department managers. The Laboratory Director also performs al documented management review annually of the quality and management systems to identify any necessary changes or improvements to the quality system or quality assurance policies.

17.0 PERSONNEL TRAINING

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at CAS are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at CAS when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at CAS. Safety training begins with the reading of the *Environmental Health and Safety Manual.* Employees are also required to attend periodic safety meetings where additional safety training may be performed by the Environmental, Health and Safety Officer. Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s).

Each employee participates in Ethics training, which is part of the CAS Improper Practices Prevention Program. CAS also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The "CAS University" education system, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

A training plan is developed for each Standard Operating Procedure. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of proficiency. Where the analyst performs the entire procedure, a generic training plan may be used. In cases where work cells are used, a training plan specific to the work cell is established.

17.1 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).

- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision. (For use of this option, LCSs must be from "second-source" standard materials independent of the calibration standards materials.).

- Where spiking is not possible but QC standards are used ("non-spiked" Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.

- Where one of the three above is not possible, special requirements are as follows:

    - Total Settleable Solids: Successful single-blind PT sample analysis and duplicate results with RPD<10%.
    - Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
    - Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 17-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

17.2 Continuing Demonstration of Proficiency

A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind PT sample analyses using the test method, or a similar test method using the same technology.

- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.

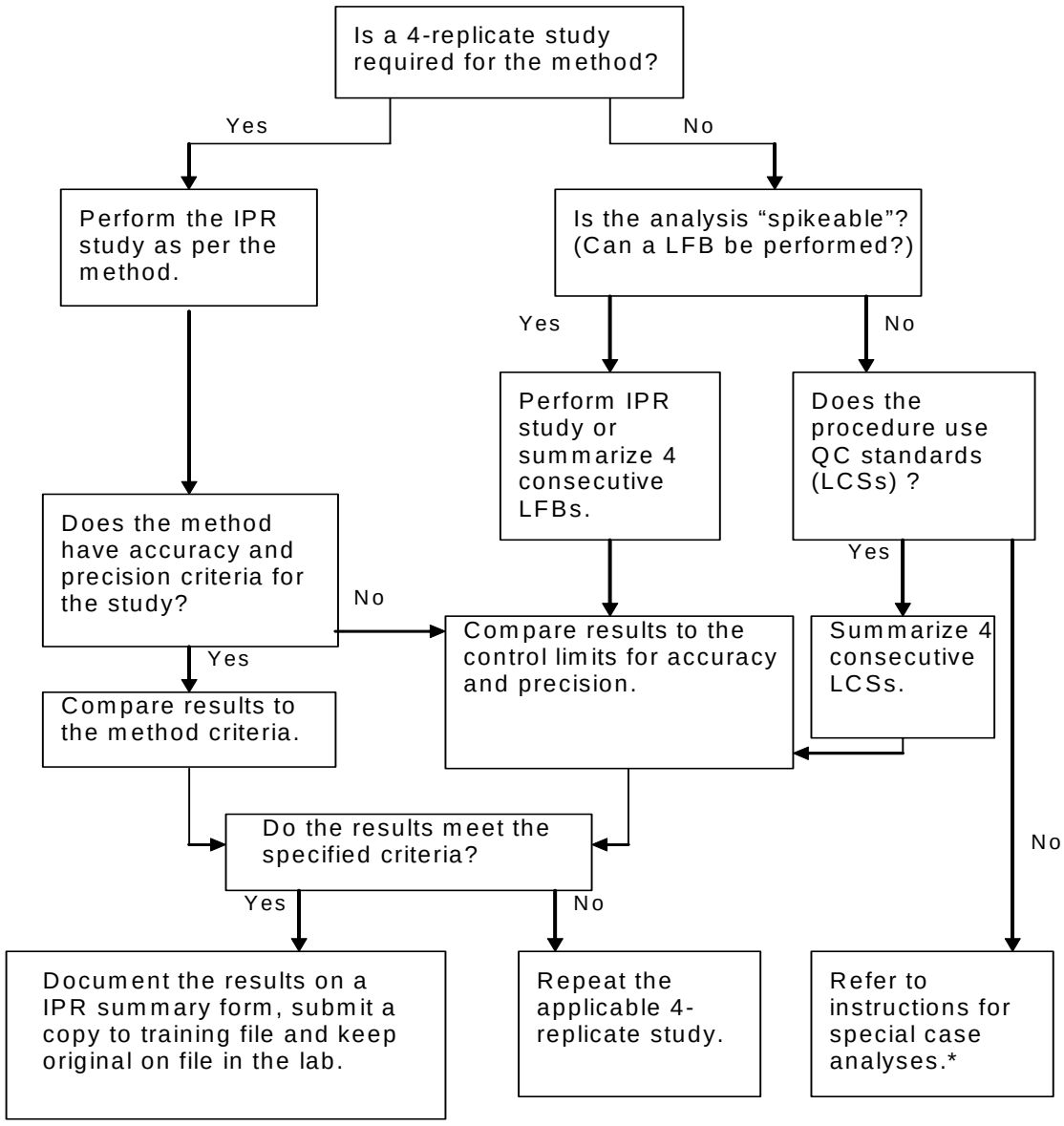
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.

- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

17.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and CAS resumes. A database is used to record the various technical skills and training acquired while employed by CAS. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Documentation of Training (SOP No. ADM-TRANDOC).*

Figure 17-1
Initial Demonstration of Capability Requirements[a]



[a] For IDOC IPR or LFB studies, "second-source" reference materials are used, as per NELAP requirements
*Total Settleable Solids: Successful PT sample analysis and duplicate results with RPD<10%.
*Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
* Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

18.0 REFERENCES FOR ANALYTICAL PROCEDURES

The analytical methods used at CAS generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at CAS are taken from the following references:

- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), Final Update IV (February 2007), and updates posted online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm. See Chapters 1, 2, 3, and 4.

- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 1983).

- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100 (August 1993).

- *Methods for the Determination of Metals in Environmental Samples*, EPA/600/4-91/010 (June 1991) and Supplements.

- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.

- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplements.

- *Standard Methods for the Examination of Water and Wastewater*, 18th Edition (1992); 19th Edition (1995), 20th Edition (1998). See Introduction in Part 1000.

- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.

- 40 CFR Part 141, National Primary Drinking Water Regulations.

- *Analytical Methods for Petroleum Hydrocarbons*, ECY 97-602, Washington State Department of Ecology, June 1997.

- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Oregon, Washington, Wisconsin, etc.).

- Annual Book of ASTM Standards, Part 31, Water.

- EPA Contract Laboratory Program, Statement of Work for Organic Analysis, SOW Nos. OLM03.1, OLM03.2, OLM04.2, and OLM04.3.

- EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, SOW No. ILM04.0, ILM04.1, and ILM05.2.

- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, EPA-540/R-94/012 (February 1993).

- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA-540/R-94/013 (February 1994).

- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994).

- *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, for USEPA and USACE (March 1986), with revisions through April 1997.

- WDOE 83-13, *Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations* (March 1982) and as Revised (July 1983 and April 1991).

- *Identification and Listing of Hazardous Waste,* California Code of Regulations, Title 22, Division 4.5, Chapter 11.

- *Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater*, EPA 821-R-93-017 (October 1993).

- *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters*, EPA 821-B-98-016 (July 1998).

- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).

- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).

- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 4th Edition, EPA 815-B-97-001 (March 1997).

- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.

- *Department of Defense Quality Systems Manual for Environmental Laboratories*, Final Version 3 (January 2006).

APPENDIX A

LIST of QA PROGRAM DOCUMENTS

Quality Assurance Manual	1/30/08
Software Quality Assurance Plan	7/11/05
CAS-Kelso Certifications/Accreditations	Cert_kel.xls
Columbia Analytical Services MDL Tracking Spreadsheet	Mdl_list.xls
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	AppSignatories.pdf
Personnel resumes/qualifications	HR Department
Personnel Job Descriptions	HR Department
Quality Control Acceptance Criteria	Qclimits.xls
Master Logbook of Laboratory Logbooks	Masterlog-001

ADMINISTRATIVE STANDARD OPERATING PROCEDURES

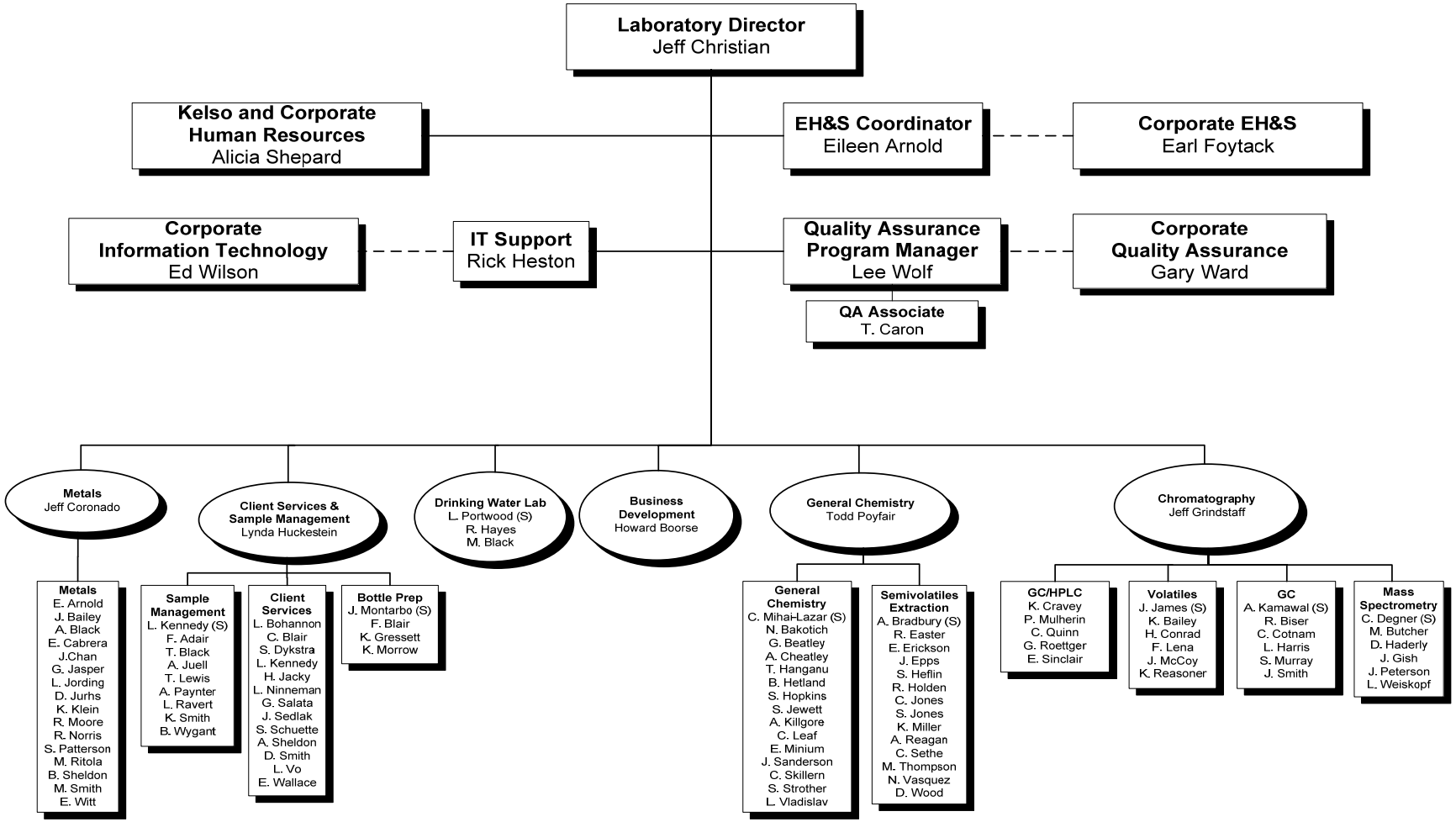
ADMINISTRATIVE - CORPORATE	**FILE NAME**
CHECKING NEW LOTS OF CHEMICALS FOR CONTAMINATION	**ADM-CTMN**
CONTROL LIMITS	**ADM-CTRL_LIM**
CORRECTIVE ACTION	**ADM-CA**
DATA RECALL	**ADM-DATARECALL**
HANDLING CUSTOMER FEEDBACK	**ADM-FDBK**
DETERMINATION OF METHOD DETECTION LIMITS AND LODS	**ADM-MDL**
DOCUMENT CONTROL	**ADM-DOCCTRL**
DOCUMENTATION OF TRAINING	**ADM-TRANDOC**
ELECTRONIC DATA AUDITING	**ADM-E_DATAAUDIT**
ESTIMATION OF UNCERTAINTY OF MEASUREMENTS	**ADM-UNCERT**
MAKING ENTRIES INTO LOGBOOKS AND ONTO BENCHSHEETS	**ADM-DATANTRY**
MANAGERIAL REVIEW OF THE LABORATORY'S QUALITY SYSTEM	**ADM-MGMTRVW**
MANUAL INTEGRATION OF CHROMATOGRAPHIC PEAKS	**ADM-INT**
PREPARATION OF ELECTRONIC DATA FOR ORGANIC ANALYSES ELECTRONIC DATA AUDITS	**ADM-EDATA**
PREPARATION OF STANDARD OPERATING PROCEDURES	**ADM-SOP**
PROFICIENCY TESTING SAMPLE ANALYSIS	**ADM-PTS**
PURCHASING THROUGH CAS PURCHASING DEPARTMENT IN KELSO	**ADM-PUR**
QUALIFICATION OF SUBCONTRACT LABORATORIES OUTSIDE OF CAS NETWORK	**ADM-SUBLAB**
SIGNIFICANT FIGURES	**ADM-SIGFIG**

ADMINISTRATIVE – LOCAL LABORATORY	**FILE NAME**
ARMY CORPS OF ENGINEERS HTRW PROJECT MANAGEMENT	**ADM-HTRW**
CHECKING PIPETTE CALIBRATION	**ADM-CPIP**
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	**ADM-ECP**
CONTROL CHARTING QUALITY CONTROL DATA	**ADM-CHRT**
DATA ARCHIVING	**ADM-ARCH**
DATA REPORTING AND REPORT GENERATION	**ADM-RG**
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	**ADM-DOD**
ELECTRONIC DATA BACKUP AND ARCHIVING	**ADM-EBACKUP**
INTERNAL QUALITY ASSURANCE AUDITS	**ADM-IAUD**
LABORATORY BALANCE MONITORING AND CALIBRATION	**ADM-BAL**
LABORATORY DATA REVIEW PROCESS	**ADM-DREV**
METHOD DETECTION LIMIT DOCUMENTATION AND CONTROL	**ADM-MDLC**
PROJECT MANAGEMENT	**ADM-PCM**
REAGENT LOGIN AND TRACKING	**ADM-RLT**
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	**ADM-SEMC**
SAMPLE MANAGEMENT SOPS	**FILE NAME**
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
SAMPLE DISPOSAL	SMO-SDIS
SAMPLE RECEIVING	SMO-GEN
SAMPLE TRACKING AND LABORATORY CHAIN OF CUSTODY	SMO-SCOC
TECHNICAL STANDARD OPERATING PROCEDURES	
SOP TABLE OF CONTENTS	SOPLIST.XLS

APPENDIX B

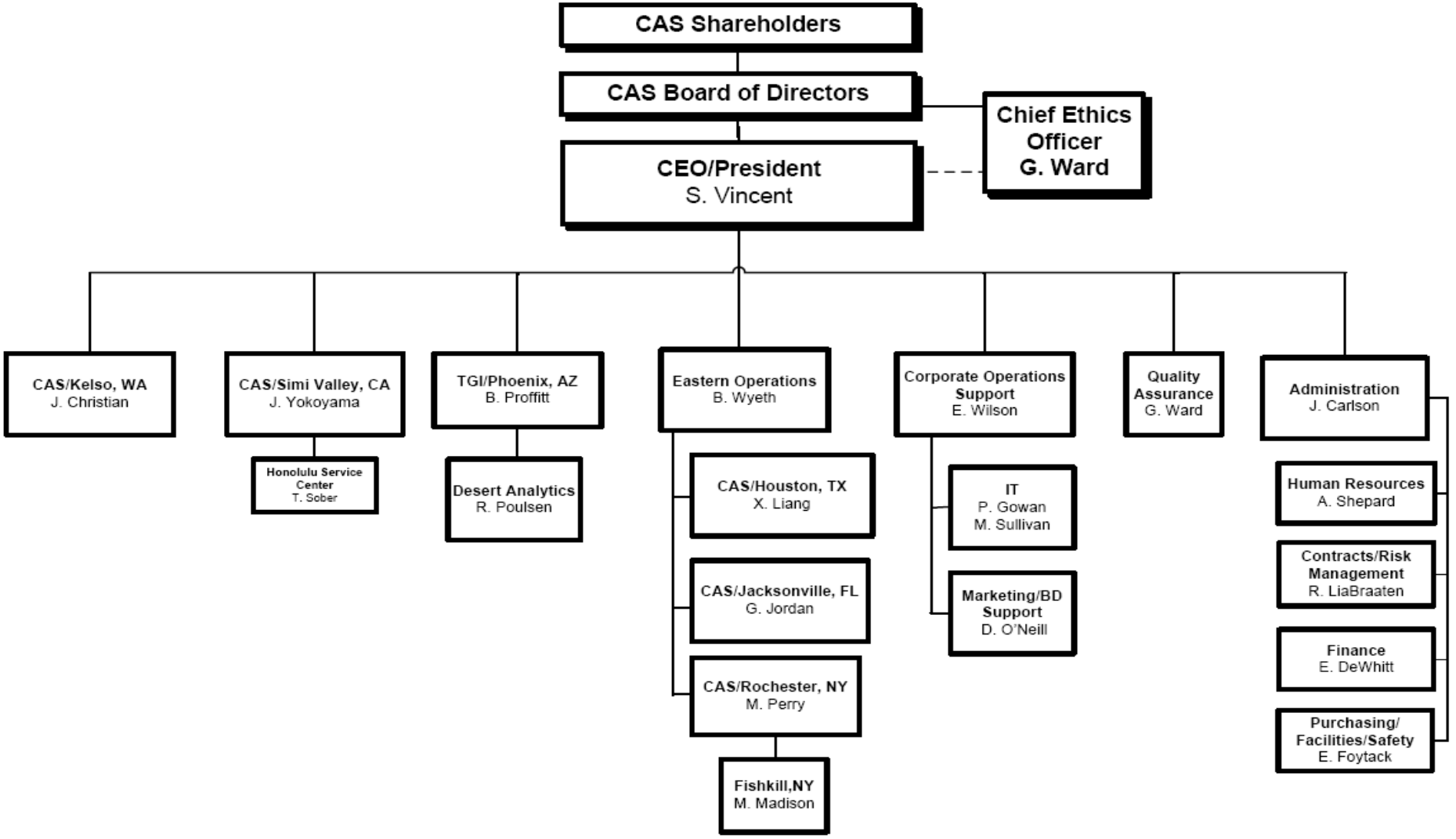
ORGANIZATIONAL CHARTS and RESUMES OF KEY PERSONNEL

Environmental and General Testing Division
Columbia Analytical Services, Inc.
Kelso, Washington Laboratory Organization



Revised 3/1/08

Columbia Analytical Services, Inc.
Laboratory Division Organization



JEFFREY D. CHRISTIAN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

VICE PRESIDENT/NW REGIONAL DIRECTOR – 1996 to Present

Responsibilities

Responsible for all phases of laboratory operations at the Kelso (WA) and Redding (CA) facilities, including project planning, budgeting, and quality assurance. Primary duties include the direct management of the Kelso laboratory (i.e. serves as the Kelso Laboratory Director, 1993-present). Also responsible for additional duties acquired as a member of the Columbia Analytical Services Holdings, Inc., Board of Directors.

Experience

Laboratory Director, Kelso Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1993-1995. Responsible for all phases of laboratory operations, including project planning, budgeting, and quality assurance.

Operations Manager, Kelso Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1993. Responsibilities included directing the daily operation of the Kelso laboratory. Other responsibilities and duties included functioning as a technical consultant to clients, providing assistance in developing and planning analytical schemes to match client objectives, and writing and developing analytical procedures/methods. Also, served as Project Manager for State of Alaska Department of Environmental Conservation contract and Coordinator for EPA Special Analytical Services (SAS) contracts.

Project Chemist and Manager, Metals Analysis Laboratory, *Columbia Analytical Services, Kelso, Washington,* 1989-1992. Responsible for directing the daily operation of the Metals Laboratory, including the sample preparation, AAS, ICP-OES, and ICP-MS Laboratories.

Scientist, *Weyerhaeuser Technology Center, Federal Way, Washington,* 1986-1989. Responsibilities included supervising atomic spectroscopy laboratory which included flame and furnace AAS, ICP-OES, and sample preparation capabilities to handle a wide variety of sample types. Interfaced with internal and external clients to provide technical support. Wrote and developed analytical procedures/methods.

Lead Technician, Metals Lab, *Weyerhaeuser Technology Center, Federal Way, Washington,* 1981-1986. Responsibilities included primary ICP and AAS analyst for EPA-CLP contract work. Extensive experience in wide variety of environmental and product-related testing.

Research Assistant, *ITT Rayonier, Olympic Research Division, Shelton, Washington,* 1978-1981. Responsibilities included performing water quality tests, product-related analytical tests, corrosion tests (i.e., potentiometric polarization techniques), and operated pilot equipment specific to the pulp and paper industry.

Education

B.S., Chemistry, *Evergreen State College, Olympia, Washington,* 1993.
ICP/MS Training Course, *VG-Elemental,* 1992.
Coursework, *Pacific Lutheran University, Tacoma, Washington.* 1988-1989.
Coursework, *Tacoma Community College, Tacoma, Washington.* 1970-1971, 1988-1989.
Perkin-Elmer Advanced Furnace, *Norwalk, Connecticut,* 1986.
CERTIFICATION, Chemistry, *L.H. Bates Technical, Tacoma, Washington,* 1978.
Coursework, *Central Washington University, Ellensburg, Washington.* 1969-1970.

Publications/ Presentations

On request.

LEE E. WOLF

1988 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER IV, KELSO QUALITY ASSURANCE MANAGER – 2002 to Present

Responsibilities

Responsible for the overall coordination of the laboratory QA program, and for ensuring that established quality objectives are met. Responsible for Quality Assurance function, including the Quality Assurance Manual, certifications, documenting SOPs, and maintaining performance evaluation records. Oversee balance calibration and sample storage temperature control. Maintain certifications/accreditations for regulatory agencies and client certifications or approval programs. Act as primary point of contact during laboratory audits. Provides audit responses and initiates any changes in procedures resulting from an audit. Coordinate the analysis of performance evaluation samples required for certification/accreditation programs. Report and review results for these analyses. Conduct internal audits and make recommendations for corrective action.

Experience

Scientist IV, Kelso Quality Assurance Manager, *Columbia Analytical Services, Inc., Kelso, Washington,* 1996-2002. Duties primarily as listed above.

Project Chemist/Principal Organic Scientist, *Columbia Analytical Services, Inc., Kelso, Washington,* 1994-1996. Responsibilities included GC and GC/MS method development and special projects coordination. Acts as technical advisor to the GC and GC/MS laboratories and GC/MS interpretation specialist and CLP organics specialist. Also responsible for Project Chemist functions, including management and coordination of projects for clients, identifying client needs, and preparation of data reports.

Semi-VOA Department Manager, *Columbia Analytical Services,* 1988-1994. Responsibilities included overall management of the Semi-VOA department. Oversee the operation of Semi-VOA GC/MS, data review and reporting and related QA/QC function. Also responsible for supervision of staff, including training, scheduling, and other personnel issues. Beginning in 1992, increased responsibilities to include Project Chemist functions for organics EPA-SAS and other clients. This involved scheduling projects for clients, identifying client needs, and preparing data reports.

GC/MS Chemist, *U.S. Testing Co., Richland, Washington,* 1985-1988. Responsibilities included GC and GC/MS analysis of water and soil samples for volatiles and Semi-VOA by EPA protocol, including Methods 8240, 8270 and CLP. Coordinated extraction and GC-GC/MS areas to manage sample/data flow through the lab. Experience also with pesticide/PCB analysis by EPA Methods 8080 and CLP. Responsible for development of analysis methods for non-routine pesticides and herbicides and performed HPLC analysis.

Laboratory Assistant, *Eastern Washington University, Cheney, Washington,* 1985. Responsibilities included supervision and instruction of organic chemistry labs. Experience with GC and IR operation. Responsible for lab safety.

Chemist Assistant, *Spokane County Air Pollution Control Authority, Spokane, Washington,* 1984. Responsibilities included gathering and analyzing air samples for CO content using IR equipment.

Education

Documenting Your Quality System, *A2LA Short Course, Las Vegas, Nevada,* 1998.
Internal Laboratory Audits, *A2LA Short Course, Las Vegas, Nevada,* 1998.
Mass Spectra Interpretation, *ACS Short Course, Denver, Colorado,* 1992.
BS, Chemistry, Minor in Geology, *Eastern Washington University, Cheney, Washington,* 1985.

Publications/ Presentations

On Request.

Affiliations

American Chemical Society.

LYNDA A. HUCKESTEIN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

CLIENT SERVICES MANAGER IV – 1998 to Present

Responsibilities

Management of the Client Services Departments: Project Management, Electronic Data Deliverables and Report Generation, and Sample Management. Personally responsible for approximately 1.5 million dollars of client work annually performing technical project management and client service. Provides technical and regulatory interpretation assistance as-well-as project organization to work received by the laboratory.

Documentation of Demonstration of Capabilities is available for review.

Experience

Project Chemist, *Columbia Analytical Service, Inc., Kelso, Washington,* 1992-1998. Primary responsibilities included technical project management and client service in areas of pulp & paper, marine services, mining, and DOD. Also responsible for providing technical and regulatory interpretation assistance as-well-as project organization to work received by the laboratory

Project Chemist and Department Manager, General Chemistry Laboratory, *Columbia Analytical Services, Inc.,* 1989-1992. Responsible for management of the General Chemistry laboratory for routine wastewater, bioassay, and microbiological analyses. Also responsible for supervision of staff, data review, and reporting.

Analyst III, *Columbia Analytical Services, Inc., Kelso, Washington,* 1989. Primary responsibilities included coliform testing, total recoverable petroleum hydrocarbon extractions and analysis, BODs, ammonias, and TKN, in addition to miscellaneous wet chemistry analyses.

Microbiologist/Chemist, *Coffey Laboratories, Portland, Oregon,* 1983. Coliform analysis; water chemistry.

Laboratory Assistant, *Oregon State University, Corvallis, Oregon,* 1983. Wheat spike dissection and tissue culture.

Education

BS, Microbiology, *Oregon State University, Corvallis, Oregon,* 1983.

JEFFREY A. CORONADO

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

Responsibilities

TECHNICAL MANAGER IV, METALS DEPARTMENT MANAGER – 2001 to Present

Primary responsibilities include management of the Metals laboratory department. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the Metals department. Also responsible for departmental budgets, method development efforts, and resource allocation.

Documentation of Demonstration of Capabilities is available for review.

Experience

Metals Department Manager, *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-2001. Responsibilities included management of all aspects of the metal laboratory operation, including personnel training and evaluation, review of all metals data, and report generation. Also responsible for client service on a number of ongoing CAS accounts. Technical duties include primary analytical responsibility for trace level metals analysis by ICP/MS. Analyses range from routine water and soil analysis, to marine tissues, as well as industrial applications such as ultra-trace QA/QC work for various semiconductor clients. Also responsible for a number of specialized sample preparation techniques including trace metals in seawater by reductive precipitation, and arsenic and selenium speciation by ion-exchange chromatography. Developed methodology for performing mercury analysis at low part per trillion levels by cold vapor atomic fluorescence..

Supervisor, GFAA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1989-1992. Responsibilities included supervision of metals analysis by graphite furnace atomic absorption following SW-846 and EPA CLP methodologies. Duties include workload scheduling, data review, instrument maintenance, personnel training and evaluation.

Education

Field Immunoassay Training Course, *EnSys Inc*., 1995.
Winter Conference on Plasma Spectrochemistry, *San Diego, California,* 1994.
ICP-MS Training Course, *VG-Elemental,* 1992.
BS, Chemistry, *Western Washington University, Bellingham, Washington,* 1988.
BA, Business Administration, *Western Washington University, Bellingham, Washington,* 1985.

JEFFREY A. GRINDSTAFF

1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER III, CHROMATOGRAPHY AND MASS SPECTROMETRY LABORATORIES – 1997 to Present

Responsibilities

Primary responsibilities include management of the GC/MS SemiVoa and VOA laboratory departments. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the GC/MS departments. Also responsible for departmental budgets, method development efforts, and resource allocation. Also performs GC/MS maintenance and troubleshooting.

Documentation of Demonstration of Capabilities is available for review.

Experience

Manager, GC/MS VOA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1994-1997. Responsible for supervision of GC/MS VOA staff, method development, training, data review, tracking department workload, scheduling analyses, and general maintenance and troubleshooting of GC/MS systems.

Scientist III, GC/MS VOA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1991-1994. Responsibilities included scheduling workload, data review, instrument maintenance and troubleshooting, and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts. Additional supervisory duties included report generation and data review for GC analyses. Responsibilities also included project management and customer service.

Chemist, *Enseco-CRL, Ventura, California,* 1990-1991. Established GC/MS department including inventory maintenance, preparation of state certification data packages, method development, SOPs, and extended data programs. Performed daily maintenance and troubleshooting of GC and GC/MS instrumentation. Scheduled and performed routine and non-routine VOA analyses.

GC/MS Chemist, VOA Laboratory *Coast-to-Coast Analytical Service, San Luis Obispo, California,* 1990-1991. Responsible for standard preparation for VOA analyses and instrument calibration, tuning, and maintenance. Also implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants..

Education

Mass Selective Detector Maintenance, *Hewlett-Packard Education Center,* 1993.
Interpretation of Mass Spectra I, *Hewlett-Packard Analytical Education Center,* 1992.
B.S., Chemistry, *California Polytechnic State University, San Luis Obispo, California,* 1989.
A.A., Liberal Arts, *Allan Hancock College, Santa Maria, California.* 1986

Publications/ Presentations

Alternate Method to Lower Detection Limits to Satisfy Regulatory Action Levels for Volatiles in Groundwater, with David Edelman, Kairas Parvez, and Paul Laymon. TAPPI National Meeting, Orlando, Florida. 1996

Affiliations

American Chemical Society. 1989

TODD N. POYFAIR

1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER III, INORGANICS & EXTRACTIONS LABORATORIES – 2001 to Present

Responsibilities

Primary responsibilities include management of the GC, HPLC, and General Chemistry laboratory departments. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the these departments. Also responsible for departmental budgets, method development efforts, and resource allocation.

Documentation of Demonstration of Capabilities is available for review.

Experience

Supervisor/Manager, General Chemistry Department, *Columbia Analytical Services, Inc., Kelso, Washington,* 1995-2001. Responsibilities included supervision, management, and training of General Chemistry staff. Also responsible for workload coordination, data review, reporting, and instrument maintenance within the General Chemistry department.

Project Chemist, Client Services Group, *Columbia Analytical Services, Inc., Kelso, Washington,* 1993-1995. Responsibilities included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to acted as liaison for all client-related activities within CAS.

Scientist II, General Chemistry Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1993. Responsibilities included the review and summarization of pH, alkalinity, conductivity, turbidity, hardness, and CODs.

Scientist I, **General Chemistry Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992. Responsibilities included analysis of Total Organic Halogens, Chemical Oxygen Demand, Sulfides, Ammonia, TKN, Nitrate/Nitrite by Lachat, and Cyanide.

Analyst III, **General Chemistry Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1991-1992. Responsibilities included analysis of pH, Conductivity, Alkalinity, Turbidity, and Oil and Grease.

Education

BS, Chemistry, *Portland State University, Portland, Oregon,* 1991.
BA, German, *Portland State University, Portland, Oregon,* 1990.
COURSEWORK, *Brigham Young University, Provo, Utah.* 1982-1983 & 1985-1986.

LOREN E. PORTWOOD

1992 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

SCIENTIST IV, DRINKING WATER LABORATORY MANAGER– 2008 to Present

Responsibilities

Responsible for the overall operation and supervision of the Organic Drinking Water department, including oversight of UCMR2 analyses. Perform analyses and conduct data review. Perform method development. Work with project management of drinking water accounts. Development of Standard Operating Procedures for drinking water methods. Operation of Varian GC/MS, Agilent GC/ECD and Agilent HPLC.

Documentation of Demonstration of Capabilities is available for review.

Experience

Scientist IV, Drinking Water Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 2002-2008. Plan, conduct, and, as lead analyst, supervise analyses using advanced instrumentation such as HPLC with post column derivatization, GC/MS, and GC/ECD. Responsible for data interpretation, QC, and data reporting. Also responsible for preparation of SOPs; handling routine and advanced maintenance and troubleshooting of instrumentation; and assisting in the training of staff department analysts. Assists the department manager and/or other senior scientists in setting up more complex procedures.

Technical Manager I, Petroleum Hydrocarbon Laboratory Supervisor, Primary responsibilities included oversight of the PHC laboratory, including initiating new processes and staff development and training. Responsible for CAS QA compliance, routine system checks. Technical mentor to PHC staff. Also duties listed below under Scientist II and Scientist III.

Scientist III, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1997-1998. Duties primarily as listed below.

Scientist II, **Petroleum Hydrocarbon Laboratory,** Columbia Analytical Services, Inc., Kelso, Washington, 1996-1997. Duties primarily as listed below, and including HPLC methods 8310, 8315, and 8330.

Scientist I, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1993-1996. Primary responsibilities included the analysis, reporting, and archiving of water, soil, and product samples for semi-volatile petroleum hydrocarbons. Methods of analysis include EPA method 8015 and various state modifications thereof (OR, WA, CA, AK). Additional responsibilities include sample preparation, instrument maintenance, and assistance with other departmental analyses.

Bench Chemist I, Organic Extractions Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1992-1993. Primary responsibilities included performing a wide range of organics extractions and cleanups for water, soil, and oil to be analyzed in the GC, GC/MS, and PHC laboratories.

Chemist, Treclen Laboratories, Spokane, Washington, 1990-1992. Primary responsibilities included inorganic water and soil testing by EPA methods. Developed testing which was accredited by the EPA, including metal digestions, phosphates, and TSS/ TDS.

Education

BS, Chemistry, Emphasis in Biochemistry, *Whitworth College, Spokane, Washington,* 1990.
Several vendor chromatography, GC, HPLC, and Quality training courses, 1993-2002.

EILEEN M. ARNOLD

1987 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

SCIENTIST IV, METALS LABORATORY, KELSO HEALTH AND SAFTEY OFFICER – 1994 to Present

Responsibilities

Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits.

Documentation of Demonstration of Capabilities is available for review.

Experience

Project Chemist, Client Services Group, Kelso Health and Safety Officer, *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1994. Duties included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within Columbia Analytical Services, Inc. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits.

Scientist IV, Metals Laboratory, Health and Safety Officer, *Columbia Analytical Services, Inc., Kelso, Washington, 1987-1992.* Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits.

Chemist, *Dow Corning Corporation, Springfield, Oregon,* 1986-1987. Responsibilities included ICP and atomic absorption work in silicon manufacturing. Methods development for ICP analysis of minor impurities found in silicon.

Chemist, *Ametek, Inc., Harleysville, Pennsylvania,* 1982-1985. Responsibilities included product research and development chemist involved in production of thin-film semiconductors for use as solar cells. Work involved AA and SEM techniques.

Chemist, *Janbridge, Inc., Philadelphia, Pennsylvania,* 1978-1982. Responsibilities included maintaining electroplating process lines through wet chemical analysis techniques, and performed Quality Assurance testing on printed circuit boards.

Education

BA, Chemistry, *Immaculata College, Immaculata, Pennsylvania,* 1977.

Affiliations

American Chemical Society, Member since 1987.

ED WILSON

1999 TO PRESENT

Columbia Analytical Services, Inc. 1317 South 13th Avenue Kelso, WA 98626 (360)577-7222

Current Position

DIRECTOR OF IT AND MARKETING – 2007 to Present

Responsibilities

Responsible for planning and implementing the CAS IT and marketing strategies.

Documentation of Demonstration of Capabilities is available for review.

Experience

Laboratory Director/Southwest Regional Manager, *Columbia Analytical Service, Inc., Canoga Park, California,* Responsible for the Canoga Park Laboratory and provides oversight of the Simi Valley, CA and Phoenix, AZ laboratories. Participates in strategic planning activities as part of the Senior Management Team of Columbia Analytical Services, Inc. 2002-2007.

Laboratory Director, *Columbia Analytical Service, Inc., Redding, California*, 1999-2002. Continued as Laboratory Director of the Redding Laboratory for Columbia Analytical Services, diversifying the laboratory into non-CH2M Hill clients.

Laboratory Director, *CH2M HILL, Redding, California*, 1998 -1999. Responsible for the operation of $3.5mm laboratory operation employing more than 40 people. Duties include P/L responsibility, and maintaining systems to ensure quality and client satisfaction. The laboratory specialized in Federal Program and Industrial Package work.

LIMS implementation, *CH2M Hill, Montgomery, Alabama,* 1997-1998. Participated as a member of the LIMS Implementation team.

Laboratory Director, *Columbia Analytical Services, Canoga Park, California,* 1996-1997. Responsible for the Southern California operation; this includes the main lab, mobile laboratories and Southern California sales. Also participates in strategic planning activities within the Southwest Division of Columbia Analytical Services, Inc.

Laboratory Director, *PACE, Camarillo, California*, 1995. Responsible for operation of the Camarillo and Fountain Valley laboratories, and the Phoenix and San Luis Obispo service centers. Also responsible for the planning, sales, marketing and financial management of the region. The laboratory specialized in Air Toxics, Sediment and Tissue analysis, and Drinking Water Analyses.

Vice President Operations, *ATI – San Diego,* 1993-1995. Responsible for ATI's eight fixed based laboratories (Anchorage, Seattle, Portland, San Diego, Phoenix, Albuquerque, Fort Collins, and Pensacola) and the nine mobile laboratories. He managed the startup of the Anchorage laboratory from writing the business plan, hiring a manager, oversight of the design and construction and staff selection through the successful startup. He served as Program Director on high profile projects such as ATI's $1M Jacobs/Navy/MCAS-Yuma mobile laboratory project. He developed and implemented systems for Variable Labor Management, Revenue and PBT Forecasting, Capacity Utilization measurement, Performance Ranking/Management Staff Planning, Job Classification, and Salary Administration. Principal role was to provide direction and advice to managers and to balance workloads and resolve resource conflicts across the network.

President, *BC Analytical*, 1980-1993. Developed a high quality, profitable well-diversified laboratory. Working with the Brown and Caldwell Consulting organization he built up the Southern California laboratories from a strong municipal wastewater business base bringing in key industrial and federal marketplace accounts. He was responsible for business development, long-range planning, budgeting staffing decisions, and overall operations for the region. He oversaw the acquisition of the PJB Laboratory from Jacobs Engineering, managed the design and construction of two laboratories - a 10,000 square-foot facility and Anaheim in 1988 and a 22,000 sq. ft. in Glendale in 1989.

Chemist, *Los Angeles Sanitation Districts, Los Angeles, California,* 1973-1980. Developed a thorough understanding of environmental chemistry, Wastewater Treatment technologies and plant operations.

Education

Accounting course work, *University of California in Los Angeles extension*, 1992.
Human Resources course work, *University of California in Los Angeles extension*, 1991.
Environmental Engineering course work, *California State University, Long Beach, California,* 1974-1976.
Post Graduate Coursework, Biochemistry, *California State University, Long Beach, California,* 1972-1976.
BA, Chemistry, *Southern Connecticut State College, New Haven, Connecticut,* 1972.

Affiliations

California State University, Long Beach, Member Curriculum Advisory Council
Society of American Military Engineers
Sierra Club

GARY K. WARD

2001 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

VICE PRESIDENT, CHIEF QUALITY, SAFETY, AND ETHICS OFFICER – 2001 to Present

Responsibilities

Responsibilities include directing and managing the overall corporate-wide quality systems, ethics and safety programs for all CAS facilities, as well as strategic planning, marketing, business development, and information technology. Responsible for all interaction and liaison with government entities involving quality, technical and operational issues.

Experience

Deputy Director, Laboratory Standards, *Intertek Testing Services, Houston, Texas,* 1998-2001. Responsibilities included professional standards/quality assurance for 240 laboratories in 93 countries, involving laboratory tests ranging from petroleum products and environmental samples to toys, textiles, and building products. Resolution of issues with a variety of governments, agiences, and companies with particular focus on interactions with the US EPA. Was previously responsible for all operations of over 100 labs in the Americas, ranging from Canada to South America, including duties to improve quality, raise profits and revenues, and implement a LIMS.

Director, Technical Operations, *Environmental Health Laboratories, South Bend, Indiana,* 1995-1998. Responsibilities included operations and quality assurance of the laboratory. Directed, administered and coordinated activities of the lab in accordance with goals and objectives of the company. Responsible for the R&D program, laboratory throughput and financial performance, and implementation of the new LIMS system.

Executive Scientist, *Quanterra (Enseco), Arvada, Colorado,* 1987-1995. Responsibilities included providing expertise and experience in laboratory analysis and operations to the entire laboratory system. Duties included implementation of network-wide LIMS as well as coordination of the Technology, QA, IS, and Operations groups. As Director of Technology and Quality Assurance was responsible for management of the R&D program, Quality Assurance program, and Environment, Health and Safety program throughout the Enseco lab system. Direct reports were all QA managers, safety managers, and chief scientists from each of the 13 laboratories.

Deputy Branch Chief, *U.S. Environmental Protection Agency,* 1983-1987. Responsibilities included providing expertise to entire Superfund program ranging from lab analytical services to sampling. Duties involved managing the CLP program as well as the Superfund R&D program. As CLP National Program Manager was responsible for development and implementation of CLP analytical protocols, administration of contracts for over 100 laboratories throughout the country, and liasion with contract divisions, other EPA programs, and enforcement. Responsible for development and implementation of disk deliverables, automated contract screening, as well as writing new protocols for specific methods such as ICP/MS and for EPA methods such as included in SW846, 3rd Edition. Duties also included coordination of the annual CLP conferences.

Education

MS, Chemical Oceanography, *RSMAS, University of Miami, Miami, Florida,* 1973.
BS, Chemistry, *Loyola University, Los Angeles, California,* 1970.

Publications, Presentations. And Affiliations

Mr. Ward has a number of publications and presentations, and is affiliated with several professional organizations. For a list of these, please contact CAS.

STEPHEN W. VINCENT

1986 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

PRESIDENT, CAS HOLDINGS INC. – 1986 to Present

Responsibilities

Responsible for the overall growth and profitability of the CAS laboratory network. This includes establishing and implementing long-range objectives, plans, and policies, and representing the company with its major customers, technical community, and the public.

Experience

Laboratory Manager, *Weyerhaeuser Company, Federal Way, Washington,* 1979-1986. Responsibilities involved all phases of technical and administrative management. This included management of organic, inorganic, and microbiological analyses and management of capital; an annual operating budget of approximately $2 million; management of thirty staff members; contract procurement, and project management. Projects included an EPA Inorganic CLP contract; an EPA acid rain deposition contract; a contract with the Fish and Wildlife Service to measure trace organic contaminants in animal tissues; and others.

Analytical Chemist, *Weyerhaeuser Company, Longview, Washington,* 1975-1979. Responsibilities: Method development, routine analysis and supervision for the Weyerhaeuser Multi-Region Support Lab. Responsible for setting up a company-wide laboratory audit, round robin, and quality assurance program.

Education

Market Strategy for Technology Based Companies, *Executives Program, Stanford University.* 1994.

Advanced Technical Management Program, *University of California at Los Angeles, Department of Business, Engineering and Management,* 1991.

Completion of Coursework for MS, Pulp and Paper Technology, *University of Washington, Seattle, Washington,* 1984.

Post Graduate Coursework, Engineering and Management, *University of California at Los Angeles, Graduate School of Engineering and Applied Science, Los Angeles, California,* 1981.

BS, Oceanography, *University of Washington, Seattle, Washington,* 1974.

Publications/ Presentations

Mr. Vincent has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.

Affiliations

American Chemical Society.
Technical Association of the Pulp and Paper Industry.

APPENDIX C

MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (9):			
Precisa and Mettler models	1988-2000	MM	15
Autoclave - Market Forge Sterilmatic	1988	LM	5
Autotitrator – Thermo Orion 500	2007	LM	3
Calorimeters (2):			
Parr 1241 EA Adiabatic	1987	LM	4
Parr 6300 Isoparabolic	2005	LM	4
Centrifuge - Damon/IEC Model K	1992	LM	15
Colony Counter - Quebec Darkfield	1988	LM	4
Conductivity Meters (2):			
YSI Model 3200	2004	LM	4
VWR	2001	LM	4
Digestion Systems (5):			
COD (4)	1987, 1989	LM	5
Kjeldahl, Lachat 46-place (1)	1999	LM	3
Dissolved Oxygen Meter - YSI Model 58 (3)	1987, 1988, 1991	LM	5
Distillation apparatus (Midi) - Easy Still (2)	1996, 2000	LM	7
Drying Ovens (11):			
Shel-Lab and VWR models	1988 - 2003	LM	15
Flash Point Testers (2):			
ERDCO Setaflash Tester	1991	LM	4
Petroleum Systems Services	2005	LM	4
Flow-Injection Analyzers (2):			
Bran-Leubbe	2002	LM	4
Lachat 8500	2007	LM	4
Ion Chromatographs (4)			
Dionex 2000i with Peaknet Data Systems	1988	LM	3
Dionex DX-120 with Peaknet Data System	1998	LM	3
Dionex ICS-2500 with Chromchem Data System	2002	LM	3
Dionex ICS-2000 with Chromchem Data System	2006	LM	3
Ion Selective Electrode Meters (5)			
Fisher Scientific Accument Model 50	1997	LM	6
Fisher Scientific Accument Model 25	1993	LM	6
Fisher Scientific Accument Model 20	2000	LM	6
Orion Model 920A	1990	LM	6
Corning pH/ion Meter Model 135	1992	LM	6
Microscope - Olympus	1988	LM	1
Muffle Furnace- Sybron Thermolyne Model F-A1730	1991	LM	15
pH Meters (2):			
Fisher Scientific Accument Model 20	1993	LM	6
Fisher Scientific Accument Model AR25	2005	LM	6

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY (continued)			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Shatter Box - GP 1000	1989	LM	5
Sieve Shakers (2):			
CE Tyler - Portable RX 24	1990	LM	5
WS Tyler - RX 86	1991	LM	5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	7
Total Organic Carbon (TOC) Analyzers (2)			
Coulemetrics Model 5012	1997	LM	3
O-I Corporation Model 1010	2002	LM	3
Total Organic Halogen (TOX) Analyzers (3):			
Mitsubishi TOX-Sigma	1995	LM	4
Mitsubishi TOX-100 (2)	2001	LM	4
Turbidimeter - Hach Model 2100N	1996	LM	8
UV-Visible Spectrophotometers (2):			
Hitachi 100-40 Single Beam	1986	LM	5
Beckman-Coulter DU520	2005	LM	5
Vacuum Pumps (2):			
Welch Duo-Seal Model 1376	1990	LM	13
Busch R-5 Series Single Stage	1991	LM	13
Water Baths/Incubators (6):			
Hach Model 15320 Incubator	1986	LM	15
Precision Model L-6 (2)	1989, 1990	LM	15
VWR 1540	1991	LM	15
Fisher 11-680-626M Incubator	1992	LM	15
Fisher Isotemp Incubator	2001	LM	15

METALS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (6)			
Mettler AE 200 analytical balance	1990	MM	12
Various Mettler, Sartorius, and Ohaus models (5)	1988	MM	12
Atomic Absorption Spectrophotometers (5):			
Varian SpectrAA Zeeman/220 AA w/Data Systems (2)	2000	LM	3
CETAC Mercury Analyzer	2000	LM	2
Perkin Elmer AAnalyst 200 Flame AA	2005	MM	2
Atomic Fluorescence Spectrophotometer			
Brooks-Rand Model III (2)	1996, 2005	LM	3
Leeman Mercury Analyzer (1)	2006	LM	2
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	12
Drying Oven - VWR Model 1370F	1990	LM	12
Freeze Dryers (2) - Labconco	1992, 2006	LM	5
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) (3)			
Thermo Jarrell Ash Model 61E	1988	LM	4
Thermo Jarrell Ash, Model IRIS	2000	MM	4
Thermo Scientific Model iCAP 6500	2007	MM	1
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES):	2000	MM	4
Inductively Coupled Plasma Mass Spectrometers (ICP-MS):			
VG PQ-S	1997	MM	3
VG Excell	2001	MM	3
Thermo X-Series	2006	MM	3
Muffle Furnace - Thermolyne Furnatrol Model 53600 (2)	1991, 2005	LM	5
Shaker - Burrell Wrist Action Model 75	1990	LM	12
TCLP Extractors (3)	1989, 2002	LM	5

SEMIVOLATILE ORGANICS SAMPLE PREPARATION LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (4)			
Mettler PM480, AE166, BB300	1999 - 2005	MM	18
OHaus EP613	2006	MM	18
Centrifuge - Sorvall Model GLC-1	1988	LM	18
Drying Ovens (2)			
Fisher Model 655G	1991	LM	18
VWR Model 1305U	1999	LM	18
Evaporators (14):			
Organomation N-Evap (7)	1989-98, 2001, 2006	LM	18
Organomation S-Evap (7)	1989-1991, 2006	LM	18
Extractor Heaters: Lab-Line Multi-Unit Models for Continuous Liquid-Liquid and Soxhlet Extractions (102)	1987-1992, 2007	LM	12
Extractors (52):			
Branson Model 450 Sonifier (2)	1991	LM	6
Tekmar Sonicator	1994	LM	6
Fisher Scientific Sonicator	1994	LM	6
Soxhtherm (48)	2000, 2008	LM	8
Extractors, TCLP (10):			
Millipore TCLP Zero Headspace Extractors (10)	1987-1992	LM	2
TCLP Extractor - Tumbler (12 position)	1989	LM	2
Gel Permeation Chromatography (GPC) (5)			
ABC single column (3)	1998, 1999, 2007	LM	4
ABC Autoprep 1000	1995	LM	4
J2 Scientific	2005	LM	4
Muffle Furnace - Parflow MIC 6000	1994	LM	4
Solid Phase Extractors (8) – Dionex SPE-Dex 4790	2003, 2006	LM	4
Ultrasonic Water Bath – VWR 550D	2007	LM	18
Vacuum Pump – Edwards	1992	LM	8

GC SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler AT 250	1989	MM	5
Chromatography Data Systems (12)			
HP Enviroquant (8)	1994-2002	LM	5
Thruput Target (4)	1998-2000	LM	5
Gas Chromatographs (14):			
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors (7)	1990 – 1995	LM	5
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FPD Detectors	1991	LM	5
Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (5)	2001, 2005, 2007	LM	5
Agilent 6890 GC with Agilent 7683 Autosampler and Dual FPD Detectors	2003	LM	5

GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Accelerated Solvent Extractor - Dionex ASE 200	1996	LM	5
HP Enviroquant Chromatography Data Systems (9)	1994-2002	LM	6
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	2
Semivolatile GC/MS Systems (9):			
Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1997, 2001	LM	5
Agilent 5890/5970 and HP 7673 Autosampler	1990	LM	5
Agilent 5890/5970 with ATAS Optic2 LVI and HP 7673 Autosampler	1994	LM	5
Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (3)	1993, 1994, 1998	LM	5
Agilent 6890/5973 with ATAS Optic3 LVI and 7683 Autosampler	2004	LM	
Agilent 6890/5973 with Agilent PTV Injector and 7683 Autosampler	2007	LM	4
Semivolatile GC/MS/MS – Waters Quattro Micro GC Micromass with Agilent 6890, Agilent PTV Injector, 7683B Autosampler	2008	MM	1

PETROLEUM HYDROCARBONS GC/HPLC LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB240	1994	MM	6
Aspirator pump – GAST	2004	LM	6
Drying Oven - Fisher Model 630F	1991	LM	6
Evaporator - Organomation N-Evap	1990	LM	6
HP Enviroquant Chromatography Data Systems (8)	1994-2002	LM	6
Gas Chromatographs (5):			
Hewlett-Packard 5890 Series II with PID/PID/FID:	1991	LM	4
Tekmar LSC-2000 Purge and Trap Concentrator	1991	LM	4
Dynatech Archon 5100 Autosampler	1992	LM	4
Hewlett-Packard 5890 GC with HP 7673 Autosampler and FID Detector	1995	LM	4
Agilent 6890 with Dual FID Detectors and Agilent 7873 Autosampler (3)	2001, 2005	LM	4
High-Performance Liquid Chromatographs (2):			
HP 1090M Series II with Diode Array UV Detector	1999	LM	4
HP 1050/1100 Series with Fluorescence & Diode Array UV Detectors	2004	LM	4
High-Performance Liquid Chromatograph/Mass Spectrometer - Thermo Electron TSQ Quantum LC/MS/MS with Thermo Surveyor HPLC and Autosampler	2005	MM	2

VOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler PE 160	1989	MM	5
Fisher Vortex Mixer	1989	LM	5
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	5
Drying Ovens (2):			
Narco 420	1989	LM	5
VWR 1305 U	1991	LM	5
Sonic Water Bath - Branson Model 2200	1989	LM	5
Volatile GC/MS Systems (7):			
Agilent 5890/5970 (2)	1989	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 5890/5971	1991	LM	5
Tekmar 3000 Purge and Trap Concentrator	2001	LM	5
Dynatech ARCHON 5100 Autosampler	1995	LM	5
Agilent 5890/5972A	1993	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 6890/5973	2001	LM	5
Tekmar 3100 Purge and Trap Concentrator	2001	LM	5
Varian Archon Autosampler	2001	LM	5
Agilent 6890/5973	2005	LM	5
Tekmar Velocity Purge and Trap Concentrator	2005	LM	5
Tekmar Aquatech Autosampler	2005	LM	5
Agilent 6890/5973	2007	LM	5
Tekmar 3000 Purge and Trap Concentrator	2007	LM	5
Varian Archon 5100 Autosampler	2007	LM	5

DRINKING WATER ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB300	1991	MM	2
Extractors (5) – Horizon SPE-DEX Solid Phase Extractor	2003	LM	2
Aglinet Enviroquant Chromatography Data Systems (2)	2003	LM	2
Varian Saturn Chromatography Data System	2003	LM	2
Evaporator - Organomation N-Evap	2003	LM	2
Agilent 1100 HPLC w/post-column derivitization:	2003	LM	2
UV/Fluoescence detectors	2003	LM	2
Pickering PCX-5200 Post-column derivitization unit	2003	LM	2
Agilent 6890N GC/ECD system:	2003	LM	2
Dual micro-ECD detectors	2003	LM	2
Agilent autosampler	2003	LM	2
Varian Ion trap GC/MS:	2003	LM	2
Varian 3800 GC w/CP8400 autosampler	2006	LM	2
Varian 3900 GC	2003	LM	2
Varian Saturn 2100T mass spectrometer	2003	LM	2

AUTOMATED DATA PROCESSING EQUIPMENT			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
1-WAN: LIMS Sample Manager using Oracle 10g DBMS running on Redhat Advanced Server 3.0 (Linux) platform connected/linked on a frame relay WAN environment	1994-2004	LM	NA
1 - Network Server Pentium 4 class, 1 for Reporting and Data Acquisition running Windows 2003 Advanced Server, 1 for Applications running Windows 2003 Advanced Server. Data acquisition capacity at 65GB with redundant tape and disk arrays.	2004	LM	NA
Approximately 50+ HP and Dell Laserjet printers (various types including models III, 4, 5, 8150, 4000, 4050, 4250, 8150, 1720dn, W5300)	1991 - 2007	LM	NA
Approximately 130 Gateway/Dell PC/Workstations running Windows 2000/XP on LAN connected via 10BT/100BT and TCP/IP for LIMs Terminal Emulation	1993 - 2004	LM	NA
Microsoft Office 2003 Professional as the base application for all PC/Workstations. Some systems using Office 2000/97.	1996 - 2004	LM	NA
E-Mail with link to SMTP for internal/external messaging. Web mail via Outlook Web Access interface. Microsoft Outlook 2003.	1994 - 2006	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Facsimile Machines - Brother 4750e (2); Brother SuperG3 (1); Canon CFX-L4000 (1)	1991 - 2007	LM	NA
Copiers/Scanners: Konica BizHub 420 (1), BizHub 600 (1), BizHub 920 (2), BizHub Pro 1050 (3). The 920s and 1050s are accessible via LAN for network scanning.	2000 - 2007	LM	NA
Dot Matrix Epson FX-880, LQ-1050, LX-300	1991 - 2004	LM	NA
Thruput, MARRS, Stealth, Harold, Blackbird, EDDGE, StarLIMS reporting software systems.	1998 - 2004	LM	NA

NA: Not applicable. This equipment administered by IT staff but may be used by all staff.

APPENDIX D

PREVENTIVE MAINTENANCE PROCEDURES

Instrument	Activity	Frequency
Refrigerators and Coolers	Record temperatures	Daily
	Clean coils	Annually
	Check coolant	Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	Every month or as needed
Fume Hoods	Face velocity measured	Quarterly
	Sash operation	As needed
	Change filters	Annually
	Inspect fan belts	Annually
Ovens	Clean	As needed or if temperature outside lim.
	Record temperatures	Daily, when in use
Incubators	Record temperatures	Daily, morning and evening
Water Baths	Record temperatures	Daily, morning and evening
	Wash with disinfectant solution	When water is murky, dirty, or growth appears
Autoclave	Check sterility	Every month
	Check temperature	Every month
	Clean	When mold or growth appears
Analytical Balances	Check alignment	Before every use
	Check calibration	Daily
	Clean pans and compartment	After every use
Dissolved Oxygen Meter	Change membrane	When fluctuations occur
pH probes	Condition probe	When fluctuations occur
Fluoride ISE	Store in storage solution	Between uses
Ammonia ISE	Store in storage solution	Between uses
UV-visible Spectrophotometer	Wavelength check	Annually
Total Organic Carbon Analyzers	Check IR zero	Weekly
	Check digestion/condensation vessels	Each use
	Clean digestion chamber	Every 2000 hours, or as needed
	Clean permeation tube	Every 2000 hours, or as needed
	Clean six-port valves	Every 200 - 2000 hours, or as needed
	Clean sample pump	Every 200 - 2000 hours, or as needed
	Clean carbon scrubber	Every 200 - 2000 hours, or as needed
	Clean IR cell	Every 2000 - 4000 hours, or as needed

Instrument	Activity	Frequency
Total Organic Halogen Analyzers	Change cell electrolyte	Daily
	Change electrode fluids	Daily
	Change pyrolysis tube	As needed
	Change inlet and outlet tubes	As needed
	Change electrodes	As needed
Flow Injection Analyzer	Check valve flares	Each use
	Check valve ports	Each use
	Check pump tubing	Each use
	Check light counts	Each use
	Check flow cell flares	Quarterly
	Change bulb	As needed
	Check manifold tubing	Each use
	Check T's and connectors	Each use
Ion Chromatographs	Change column	Every six months or as needed
	Change valve port face & hex nut	Every six months or as needed
	Clean valve slider	Every six months or as needed
	Change tubing	Annually or as needed
	Eluent pump	Annually
Atomic Absorption Spectro-photometers - FAA and CVAA	Check gases	Daily
	Clean burner head	Daily
	Check aspiration tubing	Daily
	Clean optics	Every three months
	Empty waste container	Weekly
Atomic Absorption Spectro-photometers - GFAA	Check gases	Daily
	Check argon dewar	Daily
	Change graphite tube	Daily, as needed
	Clean furnace windows	Monthly
ICP - AES	Check argon dewar	Daily
	Replace peristaltic pump tubing	Daily
	Empty waste container	Weekly
	Clean nebulizer, spray chamber, and torch	Every two weeks
	Replace water filter	Quarterly
	Replace vacuum air filters	Monthly

Instrument	Activity	Frequency
ICP - MS	Check argon dewar	Daily
	Check water level in chiller	Daily
	Complete instrument log	Daily
	Replace peristaltic pump tubing	Daily
	Clean sample and skimmer cones	As needed
	Clean RF contact strip	As needed
	Inspect nebulizer, spray chamber, and torch	Clean as needed
	Clean lens stack/extraction lens	As needed
	Check rotary pump oil	Monthly
	Change rotary pump oil	Every six months
Gel-Permeation Chromatographs	Clean and repack column	As needed
	Backflush valves	As needed
High Pressure Liquid Chromatographs	Backflush guard column	As needed
	Backflush column	As needed
	Change guard column	As needed when back pressure too high
	Change column	Annually or as needed
	Change in-line filters	As needed
	Leak check	After column maintenance
	Change pump seals	As needed
	Change pump diaphragm	Annually
	Clean flow cell	As needed
	Fluorescence detector check	Daily
	Diode array absorbance check	Daily
Gas Chromatographs, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean FID	Weekly or as needed
	Clean ECD	Quarterly or as needed
	Leak test ECD	Annually

Instrument	Activity	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Annually or as needed
	Change septum	Daily, when in use
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications
Purge and Trap Concentrators	Change trap	Every four months or as needed
	Change transfer lines	Every six months or as needed
	Clean purge vessel	Daily
Gas Chromatographs, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean PID lamp	As needed
	Clean FID	As needed
	Change ion exchange resin	Every 60 days
	Replace nickel tubing	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Annually or as needed
	Change septum	Daily
	Clip first foot of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean jet separator	As needed
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications

APPENDIX E

SOP LIST AND LIST OF NELAC ACCREDITED METHODS

COLUMBIA ANALYTICAL SERVICES, INC. , KELSO, WA.

STANDARD OPERATING PROCEDURES TABLE OF CONTENTS

SOP NAME	FILE NAME	REV #
ARMY CORPS OF ENGINEERS HTRW PROJECT MANAGEMENT	ADM-HTRW	1
CHECKING PIPETTE CALIBRATION	ADM-CPIP	4
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	ADM-ECP	1
CONTROL CHARTING QUALITY CONTROL DATA	ADM-CHRT	1
DATA ARCHIVING	ADM-ARCH	3
DATA REPORTING AND REPORT GENERATION	ADM-RG	5
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	ADM-DOD	2
ELECTRONIC DATA BACKUP AND ARCHIVING	ADM-EBACKUP	0
INTERNAL QUALITY ASSURANCE AUDITS	ADM-IAUD	6
LABORATORY BALANCE MONITORING AND CALIBRATION	ADM-BAL	0
LABORATORY DATA REVIEW PROCESS	ADM-DREV	5
METHOD DETECTION LIMIT DOCUMENTATION AND CONTROL	ADM-MDLC	2
PROJECT MANAGEMENT	ADM-PCM	8
REAGENT LOGIN AND TRACKING	ADM-RLT	2
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	ADM-SEMC	9
COLIFORM, FECAL	BIO-9221FC	5
COLIFORM, FECAL (MEMBRANE FILTER PROCEDURE)	BIO-9222D	1
COLIFORM, TOTAL	BIO-9221TC	4
COLIFORM, TOTAL (DRINKING WATER)	BIO-9221DW	4
COLILERT COMPLETED TEST VERIFICATION OF E. COLI IN MUG CULTURES	BIO-CCT	0
COLILERT	BIO-9223	4
ENTEROLERT	BIO-ENT	0
FECAL STREPTOCOCCUS/ENTEROCOCCUS	BIO-9230B	5
HEPTEROTROPHIC PLATE COUNT	BIO-HPC	3
MICROBIOLOGY QUALITY ASSURANCE AND QUALITY CONTROL	BIO-QAQC	12
SHEEN SCREEN/OIL DEGRADING MICROORGANISMS	BIO-SHEEN	1
EPA CLP ORGANICS ANALYSES	CLP_ORGA	1
ADDITION OF SPIKES AND SURROGATES	EXT-SAS	6
AUTOMATED SOXHLET EXTRACTION	EXT-3541	4
CONTINUOUS LIQUID - LIQUID EXTRACTION	EXT-3520	11
DIAZOMETHANE PREPARATION	EXT-DIAZ	5
FLORISIL CLEANUP	EXT-FLOR	2
ORGANIC EXTRACTIONS GLASSWARE CLEANING	EXT-GC	3
MEASURING SAMPLE WEIGHTS AND VOLUMES FOR ORGANIC ANALYSIS	EXT-WVOL	2
PREPARATION OF REAGENTS AND BLANK MATRICES USED IN SEMIVOLATILE ORGANICS ANALYSIS	EXT-REAG	0
REMOVAL OF SULFUR USING COPPER	EXT-3660	4
SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION	EXT-3510	7
SILICA GEL CLEANUP	EXT-3630	1
SOLID PHASE DISPERSION IN TISSUES	EXT-SPD	0
SOLID PHASE EXTRACTION	EXT-3535	3
SOXHLET EXTRACTION	EXT-3540	9
SULFURIC ACID CLEANUP	EXT-3665	2
ULTRASONIC EXTRACTION	EXT-3550	9
WASTE DILUTION EXTRACTION	EXT-3580	2
FACILITY AND LABORATORY CLEANING	FAC-CLEAN	0
OPERATION AND MAINTENANCE OF LABORATORY REAGENT WATER SYSTEMS	FAC-WATER	0
ACIDITY	GEN-305.2	3
ALKALINITY TOTAL	GEN-310.1	6
AMMONIA AS NITROGEN BY ION SPECIFIC ELECTRODE	GEN-350.3	6
AMMONIA BY FLOW INJECTION ANALYSIS	GEN-350.1	7
AUTOFLUFF	GEN-AUTOFLU	0
BIOCHEMICAL OXYGEN DEMAND	GEN-405.1	10
BULK DENSITY OF SOLID WASTE FRACTIONS	GEN-E1109	0
CARBON, TOTAL ORGANIC DETERMINATION (WALKELY BLACK METHOD)	GEN-OSU	2
CARBON, TOTAL ORGANIC IN SOIL	GEN-ASTM	6
CARBONATE (CO3) BY EVOLUTION AND COLUMETRIC TITRATION	GEN-D513-82M	0
CHEMICAL OXYGEN DEMAND	GEN-COD	7

CHLORIDE (TITRIMETRIC, MERCURIC NITRATE)	GEN-325.3	4
CHLORINE, TOTAL/FREE RESIDUAL	GEN-330-4	2
COLOR	GEN-110.2	5
COLOR, NCASI	GEN-NCAS	2
CONDUCTIVITY IN WATER AND WASTES	GEN-COND	8
CORROSIVITY TOWARDS STEEL	GEN-CORR	1
CYANIDE EXTRACTION OF SOLIDS AND OILS	GEN-9013	0
CYANIDE, WEAK ACID DISSOCIABLE	GEN-CNWAD	0
DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY	GEN-300.1	3
DISSOLVED SILICA	GEN-370.1	1
FERROUS IRON IN WATER	GEN-FeII	2
FLASHPOINT DETERMINATION - SETAFLASH	GEN-1020	6
FLUORIDE BY ION SELECTIVE ELECTRODE	GEN-FISE	5
FORMALDEHYDE COLORIMETRIC DETERMINATION	GEN-FORM	1
GLASSWASHING FOR INORGANIC ANALYSES	GEN-WASH	3
GRAVIMETRIC SULFATE	GEN-375.3	2
HALIDES, ADSORBABLE ORGANIC (AOX)	GEN-1650	2
HALIDES, ADSORBABLE ORGANIC (AOX) - SM 5320B	GEN-5320B	1
HALIDES, EXTRACTABLE ORGANIC (EOX)	GEN-9020M	2
HALIDES, TOTAL ORGANIC (TOX)	GEN-9020	8
HALOGENS TOTAL AS CHLORIDE BY BOMB COMBUSTION	GEN-5050	2
HARDNESS, TOTAL	GEN-130.2	6
HEAT OF COMBUSTION	GEN-BTU	3
HEXAVALENT CHROMIUM - COLORIMETRIC	GEN-CR6	7
HYDAZINE IN WATER USING COLORIMETRIC PROCEDURE	GEN-HYD	1
HYDROGEN HALIDES BY ION CHROMATOGTRAPHY (METHOD 26)	GEN-HA26	2
ION CHROMATOGRAPHY	GEN-IONC	11
MBAS	GEN-425.1	3
MERCURY IN COAL SAMPLE PREPARATION BY PARR BOMB COMBUSTION	GEN-HGPREP	0
NITRATE/NITRITE, NITRITE BY FLOW INJECTION ANALYSIS	GEN-353.2	6
NITRITE BY COLORIMETRIC PROCEDURE	GEN-354.1	1
NITROGEN, TOTAL AND SOLUBLE KJELDAHL	GEN-TKN	10
ORTHOPHOSPHATE BY FLOW INJECTION ANALYSIS-STANDARD METHODS 4500P-F	GEN-4500P-F	1
OXYGEN CONSUMPTION RATE	GEN-O2RATE	0
PARTICLE SIZE DETERMINATION	GEN-PSP	5
PARTICLE SIZE DETERMINATION - ASTM PROCEDURE	GEN-PSASTM	1
PERCHLORATE BY ION CHROMATOGRAPHY	GEN-314-0	12
Ph IN SOIL AND SOLIDS	GEN-Phs	9
Ph IN WATER	GEN-Phw	10
PHENOLICS, TOTAL	GEN-420.1	10
PHOSPHORUS DETERMINATION USING COLORMETRIC PROCEDURE	GEN-365.3	8
POST DIGESTION DETERMINATION OF TOTAL KJELDAHL NITROGEN BY SEMIAUTOMATED COLORIMETRY	GEN-TKNAA	0
SETTEABLE SOLIDS	GEN-160.5	4
SOLIDS, TOTAL DISSOLVED (TDS)	GEN-160.1	7
SOLIDS, TOTAL SUSPENDED (TSS)	GEN-160.2	7
SOLIDS, TOTAL VOLATILE AND PERCENT ASH IN SOIL AND SOLID SAMPLES	GEN-160.4	5
SPECIFIC GRAVITY	GEN-SPGRAV	0
SUBSAMPLING AND COMPOSITING OF SAMPLES	GEN-SUBS	3
SULFIDE, SOLUBLE DETERMINATION OF SOLUBLE SULFIDE IN SEDIMENT	GEN-DIS.S2	2
SULFIDE, TITRIMETRIC (IODINE)	GEN-376-1	1
SULFIDES, ACIDS VOLATILE	GEN-AVS	5
SULFIDE, METHYLENE BLUE	GEN-376-2	1
SULFIDES, REACTIVE	GEN-RS	4
SULFITE	GEN-SO3	2
TANNIN AND LIGNIN	GEN-5550	5
THIOCYANATE	GEN-THIOCN	0
TOTAL CYANIDES AND CYANIDES AMENABLE TO CHLORINATION	GEN-335	13
TOTAL HALIDES BY OXIDATIVE COMBUSTION AND MICROCOULOMETRY	GEN-9076	1
TOTAL ORGANIC CARBON IN WATER	GEN-TOC	9
TOTAL RESIDUAL CHLORINE - METHOD 330.5	GEN-330.5	0
TOTAL SOLIDS	GEN-160.3	11
TOTAL SULFIDE BY PSEP	GEN-S2PS	0
TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION	GEN-9030	9
TOTAL SULFUR FOR ION CHROMATOGRAPHY	GEN-ICS	1
TURBIDITY MEASUREMENT	GEN-TURB	4
ULTIMATE BOD	GEN-UBOD	0

ARSENIC BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7062	0
CATION-EXCHANGE CAPACITYOF SOILS (SODIUM ACETATE) - METHOD 9081	MET-9081	1
CLOSED VESSEL OIL DIGESTION	MET-3051M	0
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 6020)	MET-6020	11
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 200.8)	MET-ICP.MS	12
DETERMINATION OF METALS AND TRACE ELEMENTS BY ICP/AES	MET-ICP	19
DETERMINATION OF TRACE METALS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY (GFAA)	MET-GFAA	16
FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRIC ANALYSES	MET-FAA	1
MERCURY ANALYSIS BY COLD VAPOR ABSORPTION SPECTROPHOTOMETRY	MET-HG	5
MERCURY IN LIQUID WASTE	MET-7470A	11
MERCURY IN SOLID OR SEMISOLID WASTE	MET-7471A	11
MERCURY IN WATER	MET-245.1	11
MERCURY IN WATER BY OXIDATION, PURGE&TRAP, AND COLD VAPOR ATOMIC FLUORES. SPECTROMETRY	MET-1631	8
METALS AND SEMIVOLATILES TCLP EXTRACTION (EPA METHOD 1311)	MET-TCLP	6
METALS DIGESTION	MET-3005A	5
METALS DIGESTION	MET-3010A	10
METALS DIGESTION	MET-3020A	12
METALS DIGESTION	MET-3050	10
METALS DIGESTION	MET-7195	5
METALS DIGESTION - CLP	MET-DIG	9
METALS LABORATORY GLASSWARE CLEANING	MET-GC	2
METHYL MERCURY IN SOIL AND SEDIMENT BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630S	1
METHYL MERCURY IN TISSUE BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630T	0
METHYL MERCURY IN WATER BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630W	0
MULTIPLE EXTRACTION PROCEDURE	MET-MEP	0
SAMPLE FILTRATION FOR METALS ANALYSIS	MET-FILT	0
SAMPLE PREPARATION OF AQUEOUS SAMPLES BY "CLEAN" TECHNIQUES	MET-ACT	3
SAMPLE PREPARATION OF BIOLOGICAL TISSUES FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS	MET-TDIG	1
SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7742	2
TISSUE SAMPLE PREPARATION	MET-TISP	5
TRACE METALS IN WATER BY PRECONCENTRATION USING REDUCTIVE PRECIPITATION FOLLOWED BY ICP-MS	MET-RPMS	4
WASTE EXTRACTION TEST (WET) PROCEDURE (STLC) for NONVOLATILE and SEMIVOLATILE PARAMETERS	MET-STLC	0
ANALYSIS OF SOLID AND AQUEOUS SAMPLES FOR STATE OF WISCONSIN DIESEL RANGE ORGANICS	PHC-WIDRO	0
ANALYSIS OF WATER, SOLIDS AND SOLUBLE WASTE SAMPLES FOR SEMI-VOLATILE FUEL HYDROCARBONS	PET-SVF	11
GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY	PET-GRO	7
GRAVIMETRIC DETERMINATION OF HEAXANE EXTRACTABLE MATERIAL (1664)	PET-1664	5
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD	8
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT	7
SAMPLE DISPOSAL	SMO-SDIS	6
SAMPLE RECEIVING	SMO-GEN	23
SAMPLE TRACKING AND LABORATORY CHAIN OF CUSTODY	SMO-SCOC	10
ACETAMIDE HERBICIDE DEGRADATES IN DRINKING WATER BY SPE AND HPLC/MS/MS	SOC-535	0
ALDEHYDES BY HPLC	SOC-8315A	4
BUTYLTINS	SOC-BUTYL	8
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES	SOC-CAL	5
CARBON CLEANUP	SOC-CARCU	0
CHLORINATED HERBICIDES	SOC-8151	13
CHLORINATED PHENOLICS IN WASTE WATER BY IN-SITU	SOC-1653A	5
CHLORINATED PHENOLS METHOD 8151 MODIFIED	SOC-8151M	8
CONFIRMATION PROCEDURE FOR GC AND HPLC ANALYSES	SOC-CONF	4
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GG/ECD	SOC-8082C	8
DETERMINATION OF NITROGEN OR PHOSPHORUS CONTAINING PESTICIDES	SOC-8141	10
DIMP	SOC-DIMP	6
DMD SYNTHESIS	SOC-DMD	1
EXTRACTION METHOD FOR ORGANOTINS IN SEDIMENTS, WATER, AND TISSUE	SOC-OSWT	5
GEL PERMEATION CHROMATOGRAPHY	SOC-3640A	5
GLYCOLS	SOC-8015M	8
HAPS AND OTHER COMPOUNDS IN IMPINGER/CANISTER SAMPLES FROM WOOD PRODUCTS FACILITIES	SOC-9902	2
HAZARDOUS AIR POLLUTANTS (HAPS) IN PULP AND PAPER INDUSTRY CONDENSATES	SOC-9901	3
METHANOL IN PROCESS LIQUIDS	SOC-9403	4

MONOCHLOROACETIC ACID BY GC-ECD	SOC-MCA	4
NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8330	10
NITROGLYCERIN AND PETN BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8332	7
NITROGUANIDINE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-NITG	2
NONYLPHENOLS ISOMERS AND NONYLPHENOL ETHOXYLATES	SOC-NONYL	0
ORGANIC ACIDS IN AQUEOUS MATRICES BY HPLC	SOC-OALC	1
ORGANOCHLORINE PESTICIDES AND PCBs (METHOD 608)	SOC-608	5
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE	SOC-8081	10
PCBS AS AROCLORS	SOC-8082A	10
PERCENT LIPIDS IN TISSUE	SOC-LIPID	1
PESTICIDES BY GC/MS/MS	SOC-PESTMS	0
PHARMACEUTICALS, PERSONAL CARE PRODUCTS AND ENDOCRINE DISRUPTING COMPOUNDS (EDCS) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY/TANDEM MASS SPECTROMETRY (HPLC/MS/MS)	SOC-LCMS	1
PICRIC ACID AND PICRAMIC ACID BY HPLC	SOC-PICRIC	2
POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM	SOC-8270P	6
POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	SOC-8310	11
RESIN AND FATTY ACIDS BY GC/MS - NCASI METHOD 85.02 MODIFIED	SOC-85.02	0
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-625	4
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-8270C	8
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - LOW LEVEL PROCEDURE	SOC-8270L	3
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTED ION MONITORING	SOC-8270S	4
SEMI-VOLATILE ORGANICS SCREENING	SOC-SCR	2
TOTAL OLEANOLIC ACID SAPONINS IN WATER BY ACID HYDROLYSIS AND HPLC/MS/MS	SOC-LCMS3	0
1,2-DIBROMOETHANE, 1,2-DIBROMO-3-CHLOROPROPANE, AND 1,2,3-TCP BY GC	SVD-504	5
CARBAMATES AND CARBAMOYLOXIMES IN WATER BY POST-COLUMN DERIVITIZATION HPLC	SVD-531 -1	4
CHLORINATED HEBICIDES IN DRINKING WATER	SVD-515_4	5
DETERMINATION OF EXPLOSIVES AND RELATED COMPOUNDS IN DRINKING WATER BY GC/MS	SVD-529	0
DIQUAT AND PARAQUAT BY HPLC	SVD-549	4
ENDOTHALL IN DRINKING WATER BY GC/MS	SVD-548	6
GLYPHOSATE IN DRINKING WATER BY HPLC	SVD-547	4
HALOACETIC ACIDS IN DRINKING WATER	SVD-552	5
ORGANOCHLORINE PESTICIDES AND PCBS IN DRINKING WATER	SVD-508_1	4
N-NITROSAMINES BY GC/MS/MS	SVD-521	1
SELECTED PESTICIDES AND FLAME RETARDANTS IN DRINKING WATER BY GC/MS (EPA METHOD 527)	SVD-527	0
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS (METHOD 525.2)	SVD-525	5
AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 602	VOC-602BTEX	2
AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 8021	VOC-8021BTEX	4
PURGE AND TRAP FOR AQUEOUS SAMPLES	VOC-5030	4
PURGE AND TRAP/EXTRACTION FOR VOC IN SOIL AND WASTE SAMPLES , CLOSED SYSTEM	VOC-5035	7
SAMPLE SCREENING FOR VOLATILE ORGANIC COMPOUNDS IN SOIL, WATER AND MISC. MATRICES	VOC-BVOC	3
VOA STORAGE BLANKS	VOC-BLAN	5
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-524.2	10
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-624	8
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-8260	11
WA-DOH DRINKING WATER PROTOCOL	VOC-WA.DOH	2
ZERO HEADSPACE EXTRACTION (EPA METHOD 1311)	VOC-ZHE	5

Laboratory Scope of Accreditation

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State Laboratory ID: **E87412** EPA Lab Code: **WA00035** **(360) 577-7222**

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,1,1-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,1,2,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,1,2-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,1-Dichloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,1-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,1-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2,3-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2,3-Trichloropropane	EPA 504.1	Group II Unregulated Contaminants	NELAP	7/17/2003
1,2,3-Trichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,2,4-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1	Synthetic Organic Contaminants	NELAP	7/17/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1	Synthetic Organic Contaminants	NELAP	7/17/2003
1,2-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,2-Dichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,2-Dichloropropane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,3,5-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,3-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
2,2-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
2,4,5-T	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
2,4-D	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
2-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
3-Hydroxycarbofuran	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDD	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDD	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDE	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDE	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDT	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDT	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
4-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/17/2003
4-Isopropyltoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001

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Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Acetochlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Acifluorfen	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Alachlor	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Aldicarb (Temik)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldicarb sulfone	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldicarb sulfoxide	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldrin	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldrin	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Alkalinity as CaCO3	SM 2320 B	Primary Inorganic Contaminants	NELAP	10/8/2001
Aluminum	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Aluminum	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001
Antimony	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Antimony	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Arsenic	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Arsenic	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Atrazine	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Barium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Barium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Benzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Benzo(a)pyrene	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Beryllium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Beryllium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Boron	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2007
Bromate	EPA 300.1	Primary Inorganic Contaminants	NELAP	7/17/2003
Bromide	EPA 300.1	Primary Inorganic Contaminants	NELAP	7/17/2003
Bromoacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Bromobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Bromochloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Bromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/17/2003
Bromodichloromethane	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Bromoform	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Butachlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Butyl benzyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Cadmium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Cadmium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Calcium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Carbaryl (Sevin)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Carbofuran (Furaden)	EPA 531.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Carbon tetrachloride	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Chlorate	EPA 300.1	Secondary Inorganic Contaminants	NELAP	12/23/2005
Chlordane (tech.)	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Chloride	EPA 300.0	Secondary Inorganic Contaminants	NELAP	10/8/2001
Chlorite	EPA 300.1	Primary Inorganic Contaminants	NELAP	7/17/2003
Chloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Chlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Chloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Chloroform	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Chromium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Chromium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
cis-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
cis-1,3-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Color	SM 2120 B	Secondary Inorganic Contaminants	NELAP	7/17/2003
Conductivity	SM 2510 B	Primary Inorganic Contaminants	NELAP	10/8/2001
Copper	EPA 200.7	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	10/8/2001
Copper	EPA 200.8	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	10/8/2001
Copper	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Cyanide	EPA 335.4	Primary Inorganic Contaminants	NELAP	10/8/2001
Dacthal (DCPA)	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Dalapon	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
DCPA di acid degradate	EPA 515.4	Group I Unregulated Contaminants	NELAP	7/17/2003
DCPA mono acid degradate	EPA 515.4	Group I Unregulated Contaminants	NELAP	7/17/2003
Di(2-ethylhexyl)adipate	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Dibromoacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Dibromochloromethane	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Dibromomethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001

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Dicamba	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Dichloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Dichlorodifluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Dichloromethane (DCM, Methylene chloride)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Dieldrin	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Dieldrin	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Diethyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Dimethyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Di-n-butyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Di-n-octyl phthalate	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Diquat	EPA 549.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Endothall	EPA 548.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Endrin	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Endrin	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
EPTC (Eptam, s-ethyl-dipropyl thio carbamate)	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Ethylbenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Fluoride	EPA 300.0	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	10/8/2001
Fluoride	SM 4500 F-C	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	10/8/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Glyphosate	EPA 547	Synthetic Organic Contaminants	NELAP	7/17/2003
Hardness	SM 2340 B	Secondary Inorganic Contaminants	NELAP	10/8/2007
Heptachlor	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Heptachlor	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Heptachlor epoxide	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Heptachlor epoxide	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Heterotrophic plate count	SM 9215 B	Microbiology	NELAP	7/17/2003
Hexachlorobenzene	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Hexachlorobutadiene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Hexachlorocyclopentadiene	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Iron	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Isophorone	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Isopropylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Lead	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Lead	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Magnesium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Manganese	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Manganese	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001
Mercury	EPA 245.1	Primary Inorganic Contaminants	NELAP	10/8/2001
Methomyl (Lannate)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Methoxychlor	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Methoxychlor	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Methyl bromide (Bromomethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Methyl chloride (Chloromethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Methyl tert-butyl ether (MTBE)	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Metolachlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Metribuzin	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Molinate	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Molybdenum	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2007
Naphthalene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
n-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Nickel	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Nickel	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrate	EPA 300.0	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrate	EPA 353.2	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	10/8/2001
n-Propylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Orthophosphate as P	SM 4500-P F	Primary Inorganic Contaminants	NELAP	10/8/2001
Oxamyl	EPA 531.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Paraquat	EPA 549.2	Synthetic Organic Contaminants	NELAP	12/23/2005
PCBs	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Pentachlorophenol	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Pentachlorophenol	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Perchlorate	EPA 314.0	Primary Inorganic Contaminants	NELAP	7/17/2003
pH	EPA 150.1	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	10/8/2001
pH	SM 4500-H+-B	Primary Inorganic Contaminants	NELAP	4/11/2007

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-10, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87412** EPA Lab Code: **WA00035** **(360) 577-7222**

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Picloram	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Potassium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2007
Propachlor (Ramrod)	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
sec-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Selenium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Selenium	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Silica as SiO2	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Silver	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Silver	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001
Silvex (2,4,5-TP)	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Simazine	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Sodium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Styrene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Sulfate	EPA 300.0	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	10/8/2001
Terbacil	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
tert-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Tetrachloroethylene (Perchloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Thallium	EPA 200.8	Primary Inorganic Contaminants	NELAP	11/18/2004
Thallium	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Toluene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Total coliforms & E. coli	SM 9223 B	Microbiology	NELAP	10/8/2001
Total dissolved solids	SM 2540 C	Secondary Inorganic Contaminants	NELAP	10/8/2001
Total haloacetic acids	EPA 552.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Total nitrate-nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	10/8/2001
Total nitrate-nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	10/8/2001
Total organic carbon	SM 5310C	Primary Inorganic Contaminants	NELAP	4/11/2007
Total trihalomethanes	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Toxaphene (Chlorinated camphene)	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
trans-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
trans-1,3-Dichloropropylene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Trichloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Trichloroethene (Trichloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Trichlorofluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Turbidity	EPA 180.1	Secondary Inorganic Contaminants	NELAP	10/8/2001
Vanadium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2007

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Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Vinyl chloride	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Xylene (total)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Zinc	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Zinc	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001

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E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003

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E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,4,6-Tetrachlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,3-Dichlorobiphenyl (BZ 5)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001

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E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	10/8/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chlorobiphenyl (BZ 1)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	10/8/2001
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	10/8/2001
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001

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Kelso, WA 98626

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	10/8/2001
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,4,5-Trichlorocatechol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3,4,5-Trichloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3,4,6-Trichlorocatechol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3,4,6-Trichloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,5,6-Trichloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	10/8/2001
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	10/8/2001
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	10/8/2001
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 625	Extractable Organics	NELAP	10/8/2001
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Acetone	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetophenone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acidity, as CaCO3	SM 2310 B (4A)	General Chemistry	NELAP	4/11/2007
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	7/17/2003
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acrylonitrile	EPA 624	Volatile Organics	NELAP	7/17/2003
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Adsorbable organic halogens (AOX)	EPA 1650	General Chemistry	NELAP	10/8/2001
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Alkalinity as CaCO3	EPA 310.1	General Chemistry	NELAP	10/8/2001
Alkalinity as CaCO3	SM 2320 B	General Chemistry	NELAP	4/11/2007
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	12/23/2005
Aluminum	EPA 200.7	Metals	NELAP	10/8/2001
Aluminum	EPA 200.8	Metals	NELAP	10/8/2001
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Aluminum	EPA 6020	Metals	NELAP	7/1/2003
Amenable cyanide	EPA 335.1	General Chemistry	NELAP	10/8/2001
Amenable cyanide	SM 4500-CN G	General Chemistry	NELAP	4/11/2007
Ammonia as N	EPA 350.1	General Chemistry	NELAP	10/8/2001
Ammonia as N	EPA 350.3	General Chemistry	NELAP	10/8/2001
Ammonia as N	SM 4500-NH3 G	General Chemistry	NELAP	4/11/2007

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State Laboratory ID: **E87412** EPA Lab Code: **WA00035** **(360) 577-7222**

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Ammonia as N	SM 4500-NH3E	General Chemistry	NELAP	10/8/2007
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 625	Extractable Organics	NELAP	10/8/2001
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Antimony	EPA 200.7	Metals	NELAP	10/8/2001
Antimony	EPA 200.8	Metals	NELAP	10/8/2001
Antimony	EPA 6010	Metals	NELAP	7/1/2003
Antimony	EPA 6020	Metals	NELAP	7/1/2003
Aramite	EPA 8270	Extractable Organics	NELAP	7/1/2003
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Arsenic	EPA 200.7	Metals	NELAP	10/8/2001
Arsenic	EPA 200.8	Metals	NELAP	10/8/2001
Arsenic	EPA 200.9	Metals	NELAP	10/8/2001
Arsenic	EPA 6010	Metals	NELAP	10/8/2001
Arsenic	EPA 6020	Metals	NELAP	7/1/2003
Arsenic	EPA 7060	Metals	NELAP	10/8/2001
Arsenic	EPA 7062	Metals	NELAP	10/8/2007
Azinphos-methyl (Guthion)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Barium	EPA 200.7	Metals	NELAP	10/8/2001
Barium	EPA 200.8	Metals	NELAP	10/8/2001
Barium	EPA 6010	Metals	NELAP	7/1/2003
Barium	EPA 6020	Metals	NELAP	7/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzene	EPA 624	Volatile Organics	NELAP	10/8/2001
Benzene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Benzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Benzidine	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)anthracene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 200.7	Metals	NELAP	10/8/2001
Beryllium	EPA 200.8	Metals	NELAP	10/8/2001
Beryllium	EPA 6010	Metals	NELAP	7/1/2003
Beryllium	EPA 6020	Metals	NELAP	7/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
beta-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Biochemical oxygen demand	EPA 405.1	General Chemistry	NELAP	10/8/2001
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	4/11/2007
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	10/8/2001
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Bolstar (Sulprofos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Boron	EPA 200.7	Metals	NELAP	10/8/2001
Boron	EPA 6010	Metals	NELAP	10/8/2007
Bromide	EPA 300.0	General Chemistry	NELAP	10/8/2001
Bromobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromoform	EPA 624	Volatile Organics	NELAP	10/8/2001
Bromoform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Cadmium	EPA 200.7	Metals	NELAP	10/8/2001
Cadmium	EPA 200.8	Metals	NELAP	10/8/2001
Cadmium	EPA 6010	Metals	NELAP	10/8/2001
Cadmium	EPA 6020	Metals	NELAP	7/1/2003
Calcium	EPA 200.7	Metals	NELAP	10/8/2001
Calcium	EPA 6010	Metals	NELAP	10/8/2001
Carbazole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	10/8/2001
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chemical oxygen demand	EPA 410.1	General Chemistry	NELAP	10/8/2001
Chemical oxygen demand	EPA 410.2	General Chemistry	NELAP	12/23/2005
Chemical oxygen demand	SM 5220 C	General Chemistry	NELAP	4/11/2007
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloride	EPA 300.0	General Chemistry	NELAP	10/8/2001
Chloride	EPA 325.3	General Chemistry	NELAP	10/8/2001
Chloride	SM 4500 Cl- C	General Chemistry	NELAP	4/11/2007
Chlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Chloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Chloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroform	EPA 624	Volatile Organics	NELAP	10/8/2001
Chloroform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroprene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorpyrifos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chromium	EPA 200.7	Metals	NELAP	10/8/2001
Chromium	EPA 200.8	Metals	NELAP	10/8/2001
Chromium	EPA 6010	Metals	NELAP	7/1/2003
Chromium	EPA 6020	Metals	NELAP	7/1/2003
Chromium	EPA 7191	Metals	NELAP	10/8/2007
Chromium VI	EPA 7195	Metals	NELAP	7/1/2003
Chromium VI	EPA 7196	General Chemistry	NELAP	7/1/2003
Chrysene	EPA 625	Extractable Organics	NELAP	10/8/2001
Chrysene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Chrysene	EPA 8310	Extractable Organics	NELAP	7/1/2003
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	10/8/2001
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Cobalt	EPA 200.7	Metals	NELAP	10/8/2001
Cobalt	EPA 200.8	Metals	NELAP	10/8/2001
Cobalt	EPA 6010	Metals	NELAP	7/1/2003
Cobalt	EPA 6020	Metals	NELAP	7/1/2003
Color	EPA 110.2	General Chemistry	NELAP	10/8/2001
Color	SM 2120 B	General Chemistry	NELAP	4/11/2007
Conductivity	EPA 120.1	General Chemistry	NELAP	10/8/2001
Copper	EPA 200.7	Metals	NELAP	10/8/2001
Copper	EPA 200.8	Metals	NELAP	10/8/2001
Copper	EPA 6010	Metals	NELAP	10/8/2001
Copper	EPA 6020	Metals	NELAP	7/1/2003
Coumaphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Demeton-o	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diazinon	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dibenz(a,h) anthracene	EPA 625	Extractable Organics	NELAP	10/8/2001
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenz(a,h) anthracene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dibromomethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorovos (DDVP, Dichlorvos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diesel range organics (DRO)	CA-LUFT	Extractable Organics	NELAP	7/1/2003
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/28/2003
Diesel range organics (DRO)	NWTPH-Dx	Extractable Organics	NELAP	7/1/2003
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Disulfoton	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2007
Enterococci	SM 9223 B /QUANTI-TRAY	Microbiology	NELAP	10/8/2007
Escherichia coli	SM 9223 B /QUANTI-TRAY	Microbiology	NELAP	10/8/2007
Ethanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Ethoprop	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ethylbenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethylene glycol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fecal coliforms	SM 9221 E	Microbiology	NELAP	10/8/2001
Fensulfothion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fenthion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fluoranthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluoranthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 625	Extractable Organics	NELAP	10/8/2001
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Fluoride	EPA 300.0	General Chemistry	NELAP	10/8/2001
Fluoride	EPA 340.2	General Chemistry	NELAP	10/8/2001
Fluoride	SM 4500 F-C	General Chemistry	NELAP	4/11/2007
Formaldehyde	EPA 8315	Extractable Organics	NELAP	7/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	12/23/2005
Gasoline range organics (GRO)	CA-LUFT	Extractable Organics	NELAP	7/1/2003

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Kelso, WA 98626

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Gasoline range organics (GRO)	EPA 8015	Volatile Organics	NELAP	7/17/2003
Gasoline range organics (GRO)	NWTPH-Gx	Extractable Organics	NELAP	7/1/2003
Hardness	EPA 130.2	General Chemistry	NELAP	10/8/2001
Hardness	SM 2340 C	General Chemistry	NELAP	4/11/2007
Hardness (calc.)	EPA 200.7	Metals	NELAP	10/8/2007
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloroethane	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ignitability	EPA 1020	General Chemistry	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Iron	EPA 200.7	Metals	NELAP	10/8/2001
Iron	EPA 6010	Metals	NELAP	7/1/2003
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Isophorone	EPA 625	Extractable Organics	NELAP	10/8/2001
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isosafrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Kjeldahl nitrogen	ASTM D3590-89A	General Chemistry	NELAP	4/11/2007
Kjeldahl nitrogen - total	EPA 351.4	General Chemistry	NELAP	10/8/2001

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-10, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87412** EPA Lab Code: **WA00035** **(360) 577-7222**

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Lead	EPA 200.7	Metals	NELAP	10/8/2001
Lead	EPA 200.8	Metals	NELAP	10/8/2001
Lead	EPA 200.9	Metals	NELAP	10/8/2001
Lead	EPA 6010	Metals	NELAP	10/8/2001
Lead	EPA 6020	Metals	NELAP	7/1/2003
Lead	EPA 7421	Metals	NELAP	10/8/2001
Magnesium	EPA 200.7	Metals	NELAP	10/8/2001
Magnesium	EPA 6010	Metals	NELAP	7/1/2003
Malathion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Manganese	EPA 200.7	Metals	NELAP	10/8/2001
Manganese	EPA 200.8	Metals	NELAP	10/8/2001
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Manganese	EPA 6020	Metals	NELAP	7/1/2003
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Mercury	EPA 1631	Metals	NELAP	10/8/2001
Mercury	EPA 245.1	Metals	NELAP	10/8/2001
Mercury	EPA 7470	Metals	NELAP	10/8/2001
Merphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methanol	NCASI 99.01	Volatile Organics	NELAP	10/8/2001
Methanol	NCASI DI/MEOH-94.03	Volatile Organics	NELAP	10/8/2001
Methapyrilene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	10/8/2001
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	10/8/2001
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl mercury	EPA 1630	Metals	NELAP	10/8/2007
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl parathion (Parathion, methyl)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methylene chloride	EPA 624	Volatile Organics	NELAP	10/8/2001
Methylene chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Mevinphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Molybdenum	EPA 200.7	Metals	NELAP	10/8/2001
Molybdenum	EPA 200.8	Metals	NELAP	10/8/2001
Molybdenum	EPA 6010	Metals	NELAP	10/8/2001
Molybdenum	EPA 6020	Metals	NELAP	10/8/2007
Naphthalene	EPA 625	Extractable Organics	NELAP	10/8/2001
Naphthalene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Naphthalene	EPA 8310	Extractable Organics	NELAP	7/1/2003
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Nickel	EPA 200.7	Metals	NELAP	10/8/2001
Nickel	EPA 200.8	Metals	NELAP	10/8/2001
Nickel	EPA 6010	Metals	NELAP	10/8/2001
Nickel	EPA 6020	Metals	NELAP	7/1/2003
Nitrate as N	EPA 300.0	General Chemistry	NELAP	10/8/2001
Nitrate as N	EPA 353.2	General Chemistry	NELAP	7/17/2003
Nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	10/8/2001
Nitrite as N	EPA 300.0	General Chemistry	NELAP	10/8/2001
Nitrite as N	EPA 353.2	General Chemistry	NELAP	7/17/2003
Nitrobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/1/2003
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	10/8/2001
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	10/8/2001
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	10/8/2001
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/1/2003

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Oil & Grease	EPA 1664	General Chemistry	NELAP	10/8/2001
Orthophosphate as P	EPA 365.3	General Chemistry	NELAP	10/8/2001
o-Toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Oxygen, dissolved	SM 4500-O G	General Chemistry	NELAP	4/11/2007
Parathion, ethyl	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Pentachloronitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
pH	EPA 150.1	General Chemistry	NELAP	10/8/2001
pH	EPA 9040	General Chemistry	NELAP	7/1/2003
pH	SM 4500-H+-B	General Chemistry	NELAP	4/11/2007
Phenacetin	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Phenol	EPA 625	Extractable Organics	NELAP	10/8/2001
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phorate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Phosphorus, total	EPA 365.3	General Chemistry	NELAP	10/8/2001
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Potassium	EPA 200.7	Metals	NELAP	10/8/2001
Potassium	EPA 6010	Metals	NELAP	10/8/2001
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Pyrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Residual free chlorine	EPA 330.4	General Chemistry	NELAP	10/8/2001
Residue-filterable (TDS)	EPA 160.1	General Chemistry	NELAP	10/8/2001
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	4/11/2007
Residue-nonfilterable (TSS)	EPA 160.2	General Chemistry	NELAP	10/8/2001

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	4/11/2007
Residue-settleable	EPA 160.5	General Chemistry	NELAP	10/8/2001
Residue-settleable	SM 2540 F	General Chemistry	NELAP	4/11/2007
Residue-total	EPA 160.3	General Chemistry	NELAP	10/8/2001
Residue-total	SM 2540 B	General Chemistry	NELAP	4/11/2007
Residue-volatile	EPA 160.4	General Chemistry	NELAP	10/8/2001
Ronnel	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Safrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Selenium	EPA 200.7	Metals	NELAP	10/8/2001
Selenium	EPA 200.8	Metals	NELAP	10/8/2001
Selenium	EPA 200.9	Metals	NELAP	10/8/2001
Selenium	EPA 6010	Metals	NELAP	10/8/2001
Selenium	EPA 6020	Metals	NELAP	10/8/2007
Selenium	EPA 7740	Metals	NELAP	10/8/2001
Selenium	EPA 7742	Metals	NELAP	7/17/2003
Silica as SiO2	EPA 200.7	Metals	NELAP	10/8/2007
Silver	EPA 200.7	Metals	NELAP	10/8/2001
Silver	EPA 200.8	Metals	NELAP	10/8/2001
Silver	EPA 6010	Metals	NELAP	7/1/2003
Silver	EPA 6020	Metals	NELAP	7/1/2003
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Sodium	EPA 200.7	Metals	NELAP	10/8/2001
Sodium	EPA 6010	Metals	NELAP	7/1/2003
Stirofos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Strontium	EPA 200.7	Metals	NELAP	10/8/2007
Styrene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Sulfate	EPA 300.0	General Chemistry	NELAP	10/8/2001
Sulfide	EPA 376.1	General Chemistry	NELAP	10/8/2001
Sulfide	SM 4500-S F (20th Ed.)	General Chemistry	NELAP	4/11/2007
Sulfite-SO3	SM 4500-SO3 B	General Chemistry	NELAP	4/11/2007
Surfactants - MBAS	SM 5540 C	General Chemistry	NELAP	4/11/2007
Tannin & Lignin	SM 5550 B	General Chemistry	NELAP	10/8/2007
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachlorocatechol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	10/8/2001

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E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Thallium	EPA 200.7	Metals	NELAP	10/8/2001
Thallium	EPA 200.8	Metals	NELAP	10/8/2001
Thallium	EPA 200.9	Metals	NELAP	10/8/2001
Thallium	EPA 6010	Metals	NELAP	10/8/2007
Thallium	EPA 6020	Metals	NELAP	7/1/2003
Thallium	EPA 7841	Metals	NELAP	7/1/2003
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Tin	EPA 200.7	Metals	NELAP	7/17/2003
Tin	EPA 6010	Metals	NELAP	10/8/2007
Titanium	EPA 200.7	Metals	NELAP	7/17/2003
Titanium	EPA 6010	Metals	NELAP	10/8/2007
Tokuthion (Prothiophos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Toluene	EPA 624	Volatile Organics	NELAP	10/8/2001
Toluene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Toluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Total coliforms	SM 9221 B	Microbiology	NELAP	10/8/2001
Total coliforms	SM 9223 B /QUANTI-TRAY	Microbiology	NELAP	10/8/2007
Total coliforms & E. coli	SM 9223 B	Microbiology	NELAP	10/8/2007
Total cyanide	EPA 335.4	General Chemistry	NELAP	7/17/2003
Total cyanide	EPA 9012	General Chemistry	NELAP	12/23/2005
Total hardness as CaCO3	EPA 200.7	Metals	NELAP	10/8/2001
Total organic carbon	EPA 415.1	General Chemistry	NELAP	10/8/2001
Total organic carbon	EPA 9060	General Chemistry	NELAP	7/1/2003
Total organic carbon	SM 5310C	General Chemistry	NELAP	4/11/2007
Total organic halides (TOX)	EPA 9020	General Chemistry	NELAP	7/1/2003
Total Petroleum Hydrocarbons (TPH)	EPA 1664	General Chemistry	NELAP	10/8/2001
Total Petroleum Hydrocarbons (TPH)	EPA 8015	Extractable Organics	NELAP	7/1/2003
Total Petroleum Hydrocarbons (TPH)	NWTPH-HCID	Extractable Organics	NELAP	7/1/2003
Total phenolics	EPA 420.1	General Chemistry	NELAP	10/8/2001
Total residual chlorine	SM 4500-Cl F	General Chemistry	NELAP	4/11/2007
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	10/8/2001

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,3-Dichloropropylene	EPA 624	Volatile Organics	NELAP	10/8/2001
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	10/8/2001
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloronate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Trichlorosyringol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Turbidity	EPA 180.1	General Chemistry	NELAP	10/8/2001
Uranium	EPA 200.8	Metals	NELAP	10/8/2001
Vanadium	EPA 200.7	Metals	NELAP	10/8/2001
Vanadium	EPA 200.8	Metals	NELAP	10/8/2001
Vanadium	EPA 6010	Metals	NELAP	7/1/2003
Vanadium	EPA 6020	Metals	NELAP	10/8/2007
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	EPA 624	Volatile Organics	NELAP	10/8/2001
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Xylene (total)	EPA 624	Volatile Organics	NELAP	10/8/2001
Xylene (total)	EPA 8021	Volatile Organics	NELAP	7/1/2003
Xylene (total)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Zinc	EPA 200.7	Metals	NELAP	10/8/2001
Zinc	EPA 200.8	Metals	NELAP	10/8/2001
Zinc	EPA 6010	Metals	NELAP	10/8/2001
Zinc	EPA 6020	Metals	NELAP	7/1/2003

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E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	7/17/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
1-Chlorohexane	EPA 8260	Volatile Organics	NELAP	7/17/2003
1-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/17/2003
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001

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Columbia Analytical Services, Inc. - WA
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Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,3-Dichlorobiphenyl (BZ 5)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Chlorobiphenyl (BZ 1)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	10/8/2001
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	10/8/2001
2-Hexanone	EPA 8260	Volatile Organics	NELAP	10/8/2001
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Nitropropane	EPA 8260	Volatile Organics	NELAP	7/17/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	10/8/2001
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	10/8/2001
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
3-Methylphenol (m-Cresol)	EPA 8270	Extractable Organics	NELAP	7/17/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	10/8/2001
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	10/8/2001
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	10/8/2001
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	10/8/2001
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acenaphthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acenaphthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acenaphthylene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Acetone	EPA 8260	Volatile Organics	NELAP	10/8/2001
Acetonitrile	EPA 8260	Volatile Organics	NELAP	10/8/2001
Acetophenone	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	10/8/2001
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	10/8/2001
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aluminum	EPA 6010	Metals	NELAP	10/8/2001
Aluminum	EPA 6020	Metals	NELAP	10/8/2001
Ammonia as N	EPA 350.1	General Chemistry	NELAP	10/8/2007
Aniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
Anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Anthracene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Antimony	EPA 6010	Metals	NELAP	10/8/2001
Antimony	EPA 6020	Metals	NELAP	10/8/2001
Aramite	EPA 8270	Extractable Organics	NELAP	10/8/2001
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Arsenic	EPA 6010	Metals	NELAP	7/1/2003
Arsenic	EPA 6020	Metals	NELAP	10/8/2001
Arsenic	EPA 7060	Metals	NELAP	10/8/2001
Arsenic	EPA 7062	Metals	NELAP	10/8/2007

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Azinphos-methyl (Guthion)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Barium	EPA 6010	Metals	NELAP	10/8/2001
Barium	EPA 6020	Metals	NELAP	10/8/2001
Benzene	EPA 8021	Volatile Organics	NELAP	10/8/2001
Benzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(a)pyrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(b)fluoranthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(g,h,i)perylene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(k)fluoranthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzoic acid	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	10/8/2001
Beryllium	EPA 6010	Metals	NELAP	10/8/2001
Beryllium	EPA 6020	Metals	NELAP	10/8/2001
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
beta-Naphthylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	10/8/2001
Bolstar (Sulprofos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Boron	EPA 6010	Metals	NELAP	10/8/2007
Bromobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Bromoform	EPA 8260	Volatile Organics	NELAP	10/8/2001
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Cadmium	EPA 6010	Metals	NELAP	10/8/2001
Cadmium	EPA 6020	Metals	NELAP	10/8/2001
Calcium	EPA 6010	Metals	NELAP	10/8/2001
Carbazole	EPA 8270	Extractable Organics	NELAP	10/8/2001

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Carbon disulfide	EPA 8260	Volatile Organics	NELAP	10/8/2001
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Chloride	EPA 300.0	General Chemistry	NELAP	2/17/2006
Chloride	EPA 9056	General Chemistry	NELAP	7/17/2003
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Chloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chloroform	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chloroprene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chlorpyrifos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Chromium	EPA 6010	Metals	NELAP	10/8/2001
Chromium	EPA 6020	Metals	NELAP	10/8/2001
Chromium	EPA 7191	Metals	NELAP	10/8/2007
Chromium VI	EPA 7195	Metals	NELAP	10/8/2001
Chromium VI	EPA 7196	General Chemistry	NELAP	10/8/2001
Chrysene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Chrysene	EPA 8310	Extractable Organics	NELAP	10/8/2001
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	10/8/2001
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Cobalt	EPA 6010	Metals	NELAP	10/8/2001
Cobalt	EPA 6020	Metals	NELAP	10/8/2001
Copper	EPA 6010	Metals	NELAP	10/8/2001
Copper	EPA 6020	Metals	NELAP	10/8/2001
Corrosivity (pH)	EPA 1110	General Chemistry	NELAP	10/8/2001
Coumaphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Demeton-o	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Demeton-s	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Diazinon	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Dibenz(a,h) anthracene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	10/8/2001

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Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Dibromomethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dichlorovos (DDVP, Dichlorvos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Diesel range organics (DRO)	CA-LUFT	Extractable Organics	NELAP	10/8/2001
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/17/2003
Diesel range organics (DRO)	NWTPH-Dx	Extractable Organics	NELAP	10/8/2001
Diethyl ether	EPA 8260	Volatile Organics	NELAP	7/17/2003
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Dimethoate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8270	Extractable Organics	NELAP	10/8/2001
Disulfoton	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2007
EPN	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Ethoprop	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	7/17/2003
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/8/2001
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	10/8/2001
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Ethylene glycol	EPA 8015	Volatile Organics	NELAP	10/8/2001
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001

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State Laboratory ID: **E87412** EPA Lab Code: **WA00035** **(360) 577-7222**

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Fensulfothion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Fenthion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Fluoranthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Fluoranthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Fluorene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Fluorene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Fluoride	EPA 300.0	General Chemistry	NELAP	2/17/2006
Fluoride	EPA 9056	General Chemistry	NELAP	7/17/2003
Formaldehyde	EPA 8315	Extractable Organics	NELAP	10/8/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Gasoline range organics (GRO)	CA-LUFT	Extractable Organics	NELAP	10/8/2001
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	7/17/2003
Gasoline range organics (GRO)	NWTPH-Gx	Extractable Organics	NELAP	10/8/2001
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Ignitability	EPA 1020	General Chemistry	NELAP	10/8/2001
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Indeno(1,2,3-cd)pyrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Iron	EPA 6010	Metals	NELAP	10/8/2001
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Isophorone	EPA 8270	Extractable Organics	NELAP	10/8/2001
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Isosafrole	EPA 8270	Extractable Organics	NELAP	10/8/2001
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Lead	EPA 6010	Metals	NELAP	10/8/2001
Lead	EPA 6020	Metals	NELAP	10/8/2001

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Lead	EPA 7421	Metals	NELAP	10/8/2001
Magnesium	EPA 6010	Metals	NELAP	10/8/2001
Malathion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Manganese	EPA 6010	Metals	NELAP	10/8/2001
Manganese	EPA 6020	Metals	NELAP	10/8/2001
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Mercury	EPA 1631	Metals	NELAP	10/8/2007
Mercury	EPA 7470	Metals	NELAP	10/8/2001
Mercury	EPA 7471	Metals	NELAP	10/8/2001
Merphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methapyrilene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methyl mercury	EPA 1630	Metals	NELAP	10/8/2007
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/17/2003
Methyl parathion (Parathion, methyl)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methylene chloride	EPA 8260	Volatile Organics	NELAP	10/8/2001
Mevinphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Molybdenum	EPA 6010	Metals	NELAP	10/8/2001
Molybdenum	EPA 6020	Metals	NELAP	10/8/2007
Naphthalene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Naphthalene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Naphthalene	EPA 8310	Extractable Organics	NELAP	10/8/2001
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Nickel	EPA 6010	Metals	NELAP	10/8/2001
Nickel	EPA 6020	Metals	NELAP	10/8/2001
Nitrate	EPA 9056	General Chemistry	NELAP	7/17/2003
Nitrate as N	EPA 353.2	General Chemistry	NELAP	10/8/2007
Nitrite	EPA 9056	General Chemistry	NELAP	7/17/2003
Nitrite as N	EPA 353.2	General Chemistry	NELAP	10/8/2007

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	10/8/2001
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	7/17/2003
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	10/8/2001
Oil & Grease	EPA 1664	General Chemistry	NELAP	10/8/2001
Oil & Grease	EPA 9071	General Chemistry	NELAP	10/8/2001
Orthophosphate as P	EPA 365.3	General Chemistry	NELAP	10/8/2007
o-Toluidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
Parathion, ethyl	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
p-Dioxane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/17/2003
Pentachloronitrobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
pH	EPA 9040	General Chemistry	NELAP	10/8/2001
pH	EPA 9045	General Chemistry	NELAP	7/17/2003
Phenacetin	EPA 8270	Extractable Organics	NELAP	10/8/2001
Phenanthrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Phenanthrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Phenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
Phorate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Phosphorus, total	EPA 365.3	General Chemistry	NELAP	10/8/2007
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	10/8/2001

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Potassium	EPA 6010	Metals	NELAP	10/8/2001
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	10/8/2001
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Pyrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Pyrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Pyridine	EPA 8270	Extractable Organics	NELAP	10/8/2001
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	10/8/2001
Residue-total	EPA 160.3	General Chemistry	NELAP	10/8/2007
Ronnel	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Safrole	EPA 8270	Extractable Organics	NELAP	10/8/2001
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Selenium	EPA 6010	Metals	NELAP	7/17/2003
Selenium	EPA 6020	Metals	NELAP	10/8/2007
Selenium	EPA 7740	Metals	NELAP	10/8/2001
Selenium	EPA 7742	Metals	NELAP	7/17/2003
Silver	EPA 6010	Metals	NELAP	10/8/2001
Silver	EPA 6020	Metals	NELAP	10/8/2001
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Sodium	EPA 6010	Metals	NELAP	10/8/2001
Stirofos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Strontium	EPA 6010	Metals	NELAP	10/8/2007
Strontium	EPA 6020	Metals	NELAP	10/8/2007
Styrene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Sulfate	EPA 300.0	General Chemistry	NELAP	2/17/2006
Sulfate	EPA 9056	General Chemistry	NELAP	7/17/2003
Sulfide	EPA 9030/9034	General Chemistry	NELAP	7/17/2003
Sulfotepp	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	7/17/2003
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	7/17/2003
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	10/8/2001
Thallium	EPA 6010	Metals	NELAP	7/17/2003
Thallium	EPA 6020	Metals	NELAP	10/8/2001
Thallium	EPA 7841	Metals	NELAP	10/8/2001
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Tin	EPA 6010	Metals	NELAP	10/8/2007
Tokuthion (Prothiophos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Toluene	EPA 8021	Volatile Organics	NELAP	10/8/2001
Toluene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Total cyanide	EPA 9012	General Chemistry	NELAP	12/23/2005
Total nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	10/8/2007
Total organic carbon	EPA 9060	General Chemistry	NELAP	10/8/2001
Total organic halides (TOX)	EPA 9020	General Chemistry	NELAP	10/8/2001
Total Petroleum Hydrocarbons (TPH)	NWTPH-HCID	Extractable Organics	NELAP	10/8/2001
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	10/8/2001
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Trichloronate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Vanadium	EPA 6010	Metals	NELAP	10/8/2001
Vanadium	EPA 6020	Metals	NELAP	10/8/2007
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	10/8/2001
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	10/8/2001
Xylene (total)	EPA 8021	Volatile Organics	NELAP	10/8/2001
Xylene (total)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Zinc	EPA 6010	Metals	NELAP	10/8/2001
Zinc	EPA 6020	Metals	NELAP	10/8/2001

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Matrix: **Biological Tissue**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3-Dichlorobiphenyl (BZ 5)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003

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Matrix: **Biological Tissue**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Chlorobiphenyl (BZ 1)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Aluminum	EPA 6020	Metals	NELAP	7/1/2003
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Antimony	EPA 6010	Metals	NELAP	7/1/2003
Antimony	EPA 6020	Metals	NELAP	7/1/2003

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State Laboratory ID: **E87412** EPA Lab Code: **WA00035** **(360) 577-7222**

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: **Biological Tissue**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Arsenic	EPA 6010	Metals	NELAP	7/1/2003
Arsenic	EPA 6020	Metals	NELAP	7/1/2003
Arsenic	EPA 7060	Metals	NELAP	7/1/2003
Barium	EPA 6010	Metals	NELAP	7/1/2003
Barium	EPA 6020	Metals	NELAP	7/1/2003
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 6010	Metals	NELAP	7/1/2003
Beryllium	EPA 6020	Metals	NELAP	7/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Boron	EPA 6010	Metals	NELAP	10/8/2007
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Cadmium	EPA 6010	Metals	NELAP	7/1/2003
Cadmium	EPA 6020	Metals	NELAP	7/1/2003
Carbazole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chromium	EPA 6010	Metals	NELAP	7/1/2003
Chromium	EPA 6020	Metals	NELAP	7/1/2003
Chromium	EPA 7191	Metals	NELAP	10/8/2007
Chromium VI	EPA 7196	Metals	NELAP	7/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Chrysene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Cobalt	EPA 6010	Metals	NELAP	7/1/2003
Cobalt	EPA 6020	Metals	NELAP	7/1/2003
Copper	EPA 6010	Metals	NELAP	7/1/2003
Copper	EPA 6020	Metals	NELAP	7/1/2003
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Iron	EPA 6010	Metals	NELAP	7/1/2003
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Lead	EPA 6010	Metals	NELAP	7/1/2003
Lead	EPA 6020	Metals	NELAP	7/1/2003
Lead	EPA 7421	Metals	NELAP	7/1/2003
Manganese	EPA 6010	Metals	NELAP	7/1/2003

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Laboratory Scope of Accreditation

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Manganese	EPA 6020	Metals	NELAP	7/1/2003
Mercury	EPA 1631	Metals	NELAP	10/8/2007
Mercury	EPA 7471	Metals	NELAP	7/1/2003
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Molybdenum	EPA 6010	Metals	NELAP	7/1/2003
Molybdenum	EPA 6020	Metals	NELAP	10/8/2007
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Nickel	EPA 6010	Metals	NELAP	7/1/2003
Nickel	EPA 6020	Metals	NELAP	7/1/2003
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/1/2003
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Selenium	EPA 6010	Metals	NELAP	7/1/2003
Selenium	EPA 6020	Metals	NELAP	10/8/2007
Selenium	EPA 7740	Metals	NELAP	7/1/2003
Selenium	EPA 7742	Metals	NELAP	7/1/2003
Silver	EPA 6010	Metals	NELAP	7/1/2003
Silver	EPA 6020	Metals	NELAP	7/1/2003
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Thallium	EPA 6020	Metals	NELAP	7/1/2003
Thallium	EPA 7841	Metals	NELAP	7/1/2003
Tin	EPA 6010	Metals	NELAP	10/8/2007
Total cyanide	EPA 9012	General Chemistry	NELAP	12/23/2005
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Vanadium	EPA 6010	Metals	NELAP	7/1/2003
Vanadium	EPA 6020	Metals	NELAP	10/8/2007
Zinc	EPA 6010	Metals	NELAP	7/1/2003

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Matrix: **Biological Tissue**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Zinc	EPA 6020	Metals	NELAP	7/1/2003

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APPENDIX F

ADDITIONAL AGENCY-SPECIFIC DOCUMENTS

GENERAL CHEMISTRY/WATER CHEMISTRY ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
1650C	NA	Water	Absorbable Organic Halides	10	4	ug/L
305.1/SM 2310B 4a	NA	Water	Acidity as CaCO3	2	-	mg/L
310.1 / SM 2320B	NA	Water	Alkalinity as CaCO3	2	1	mg/L
350.1M	SOP	Soil	Ammonia as Nitrogen	0.5	0.04	mg/kg
350.1	NA	Water	Ammonia as Nitrogen	0.05	0.008	mg/L
350.3/SM 4500NH3 G	NA	Water	Ammonia as Nitrogen	0.05	0.02	mg/L
405.1/SM 5210B	NA	Water	Biological Oxygen Demand	4	0.4	mg/L
300.1	NA	Water	Bromate	5	2	ug/L
300.0	NA	Water	Bromide	0.1	0.006	mg/L
300.1	NA	Water	Bromide	20	5	ug/L
410.1 and 410.2M	SOP	Soil	Chemical Oxygen Demand	200	-	mg/kg
410.1/410.2/SM 5220C	NA	Water	Chemical Oxygen Demand	5	3	mg/L
300.0M	SOP	Soil	Chloride	1	0.4	mg/kg
300.0	NA	Water	Chloride	0.2	0.008	mg/L
325.3/SM 4500Cl- C/9252	NA	Water	Chloride, Titrimetric	0.5	0.2	mg/L
330.4/SM 4500 Cl- F	NA	Water	Chlorine, Total Residual	0.1	0.05	mg/L
330.5/SM 4500 Cl- G	NA	Water	Chlorine, Total Residual	0.05	0.004	mg/L
300.1	NA	Water	Chlorite	20	2	ug/L
110.2/SM 2120B	NA	Water	Color	NA	NA	Color
NCASI	NA	Water	Color	5	2	Color
120.1 / SM 2510B	NA	Water	Conductivity	2	0.4	umhos/cm
1110	NA	Liquid	Corrosivity	NA	NA	mm/yr
9040	NA	Water	Corrosivity (pH)	NA	NA	pH units
335.4	NA	Water	Cyanide, Total	0.01	0.002	mg/L
9010B/9012A	SOP	Soil	Cyanide, Total and Amenable	0.2	-	mg/kg
9010B/9012A	NA	Water	Cyanide, Total and Amenable	0.01	0.002	mg/L
335.1M	SOP	Soil	Cyanides Amenable to Chlorination	0.2	-	mg/kg
335.1/SM 4500CN G	NA	Water	Cyanides Amenable to Chlorination	0.01	0.002	mg/L
1020.0	NA	Soil	Flashpoint, Setaflash	NA	NA	Deg. F
1020.0	NA	Water	Flashpoint, Setaflash	NA	NA	Deg. F
300.0M	SOP	Soil	Fluoride	1	0.2	mg/kg

GENERAL CHEMISTRY/WATER CHEMISTRY ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
300.0	NA	Water	Fluoride	0.2	0.005	mg/L
340.2M	SOP	Soil	Fluoride	40	-	mg/kg
340.2 / SM 4500-F-C	NA	Water	Fluoride	0.2	0.009	mg/L
340.1M	SOP	Soil	Fluoride, Bellack Distillation	1	0.2	mg/kg
340.1	NA	Water	Fluoride, Bellack Distillation	1	0.2	mg/L
130.2 / SM 2340C	NA	Water	Hardness as CaCO3	2	0.9	mg/L
7196A	3060A	Soil	Hexavalent Chromium	0.5	0.06	mg/kg
7196A	Method	Water	Hexavalent Chromium	0.05	0.02	mg/L
300.0M	SOP	Soil	Nitrate as Nitrogen	0.1	0.004	mg/kg
300.0	NA	Water	Nitrate as Nitrogen	0.1	0.004	mg/L
300.0M	SOP	Soil	Nitrite as Nitrogen	0.1	0.002	mg/kg
300.0	NA	Water	Nitrite as Nitrogen	0.1	0.002	mg/L
354.1/SM 4500NO2 B	NA	Water	Nitrite as Nitrogen, Colorimetric	0.01	0.002	mg/L
353.2M	SOP	Soil	Nitrogen, Nitrate + Nitrite as Nitrogen	0.5	0.04	mg/kg
353.2	NA	Water	Nitrogen, Nitrate + Nitrite as Nitrogen	0.05	0.005	mg/L
351.4M	SOP	Soil	Nitrogen, Total Kjeldahl	20	5	mg/kg
351.4/ASTM D3590-89A	NA	Water	Nitrogen, Total Kjeldahl	0.2	0.07	mg/L
365.3M	SOP	Soil	Orthophosphate as Phosphorus	0.1	-	mg/kg
365.3	NA	Water	Orthophosphate as Phosphorus	0.01	0.003	mg/L
SM 4500P-E	NA	Water	Orthophosphate as Phosphorus	0.05	0.02	mg/L
SM 4500P-F	NA	Water	Orthophosphate as Phosphorus	0.05	0.03	mg/L
ASTM D1498	NA	Water	Oxidation-Reduction Potential	NA	NA	mV
9095.0	NA	Soil	Paint Filter Test	NA	NA	NA
314.0	NA	Water	Perchlorate	1	0.4	ug/L
150.1/SM 4500H+ B	NA	Water	pH	NA	NA	pH units
9045C	NA	Soil	pH	NA	NA	pH units
420.1M	SOP	Soil	Phenolics, Total	0.05	-	mg/kg
420.1	NA	Water	Phenolics, Total	0.01	0.009	mg/L
365.3M	SOP	Soil	Phosphorus, Total	0.1	-	mg/kg
365.3	NA	Water	Phosphorus, Total	0.01	0.004	mg/L
160.5/SM 2540F	NA	Water	Solids, Settleable	0.1	-	mg/L
160.3M	NA	Soil	Solids, Total	NA	NA	%

GENERAL CHEMISTRY/WATER CHEMISTRY ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
160.3/SM 2540B	NA	Water	Solids, Total	5	NA	mg/L
160.1 / SM 2540C	NA	Water	Solids, Total Dissolved (Filterable)	5	NA	mg/L
160.2/SM 2540D	NA	Water	Solids, Total Suspended (Nonfilterable)	5	NA	mg/L
160.4	NA	Soil	Solids, Volatile	NA	NA	%
160.4	NA	Water	Solids, Volatile	NA	NA	mL/L
300.0M	SOP	Soil	Sulfate	1	-	mg/kg
300.0	NA	Water	Sulfate	0.2	0.006	mg/L
376.1/SM 4500S2- F	NA	Water	Sulfide	2	0.4	mg/L
376.2/SM 4500S2- D	NA	Water	Sulfide	0.05	0.003	mg/L
9030M	Method	Sediment	Sulfides	0.5	0.05	mg/kg
PSEP	PSEP	Sediment	Sulfides	1	0.6	mg/kg
821/R-91-100 Draft	Method	Sediment	Acid Volatile Sulfides	0.5	0.09	mg/kg
9030	NA	Water	Sulfides	0.1	0.02	mg/L
9030	SOP	Soil	Sulfides, Acid Volatile	0.5	0.09	mg/kg
377.1/SM 4500SO32-B	NA	Water	Sulfite	2	0.3	mg/L
425.1/SM 5540C	NA	Water	Surfactants (MBAS)	0.05	0.05	mg/L
SM 5550B	NA	Water	Tannin and Lignin	0.2	0.05	mg/L

METALS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
1631	Method	Soil	Mercury	1	0.2	ug/kg
7471A	Method	Soil	Mercury	0.02	0.002	mg/kg
245.1	Method	Water	Mercury	0.2	0.05	ug/L
1631	Method	Water	Mercury	1	0.08	ng/L
7470A	Method	Water	Mercury	0.2	0.05	ug/L
CAS SOP	Method	Soil	Methyl Mercury	0.4	0.04	ng/g
1630	Method	Water	Methyl Mercury	0.1	0.05	ng/L
CAS SOP	Method	Tissue	Methyl Mercury	10	5	ng/g
200.7 (ICP)	Method	Soil	Aluminum	10	9	mg/kg
200.7 (ICP)	Method	Soil	Antimony	10	4	mg/kg
200.7 (ICP)	Method	Soil	Arsenic	20	6	mg/kg
200.7 (ICP)	Method	Soil	Barium	2	0.3	mg/kg
200.7 (ICP)	Method	Soil	Beryllium	1	0.04	mg/kg
200.7 (ICP)	Method	Soil	Boron	10	3	mg/kg
200.7 (ICP)	Method	Soil	Cadmium	1	0.4	mg/kg
200.7 (ICP)	Method	Soil	Calcium	10	4	mg/kg
200.7 (ICP)	Method	Soil	Chromium	2	0.7	mg/kg
200.7 (ICP)	Method	Soil	Cobalt	2	0.7	mg/kg
200.7 (ICP)	Method	Soil	Copper	2	0.9	mg/kg
200.7 (ICP)	Method	Soil	Iron	4	2	mg/kg
200.7 (ICP)	Method	Soil	Lead	20	3	mg/kg
200.7 (ICP)	Method	Soil	Lithium	2	1	mg/kg
200.7 (ICP)	Method	Soil	Magnesium	4	0.8	mg/kg
200.7 (ICP)	Method	Soil	Manganese	2	0.2	mg/kg
200.7 (ICP)	Method	Soil	Molybdenum	2	0.7	mg/kg
200.7 (ICP)	Method	Soil	Nickel	4	2	mg/kg
200.7 (ICP)	Method	Soil	Phosphorus	40	30	mg/kg
200.7 (ICP)	Method	Soil	Potassium	200	200	mg/kg
200.7 (ICP)	Method	Soil	Selenium	40	6	mg/kg
200.7 (ICP)	Method	Soil	Silver	2	0.9	mg/kg

METALS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
200.7 (ICP)	Method	Soil	Sodium	20	20	mg/kg
200.7 (ICP)	Method	Soil	Strontium	2	0.2	mg/kg
200.7 (ICP)	Method	Soil	Thallium	40	8	mg/kg
200.7 (ICP)	Method	Soil	Tin	10	2	mg/kg
200.7 (ICP)	Method	Soil	Titanium	2	0.5	mg/kg
200.7 (ICP)	Method	Soil	Vanadium	2	0.7	mg/kg
200.7 (ICP)	Method	Soil	Zinc	2	0.4	mg/kg
200.7 (ICP)	Method	Water	Aluminum	50	40	ug/L
200.7 (ICP)	Method	Water	Antimony	50	20	ug/L
200.7 (ICP)	Method	Water	Arsenic	100	30	ug/L
200.7 (ICP)	Method	Water	Barium	5	0.9	ug/L
200.7 (ICP)	Method	Water	Beryllium	5	0.2	ug/L
200.7 (ICP)	Method	Water	Boron	50	8	ug/L
200.7 (ICP)	Method	Water	Cadmium	5	4	ug/L
200.7 (ICP)	Method	Water	Calcium	50	30	ug/L
200.7 (ICP)	Method	Water	Chromium	5	3	ug/L
200.7 (ICP)	Method	Water	Cobalt	10	5	ug/L
200.7 (ICP)	Method	Water	Copper	10	4	ug/L
200.7 (ICP)	Method	Water	Iron	20	5	ug/L
200.7 (ICP)	Method	Water	Lead	50	30	ug/L
200.7 (ICP)	Method	Water	Lithium	10	6	ug/L
200.7 (ICP)	Method	Water	Magnesium	20	4	ug/L
200.7 (ICP)	Method	Water	Manganese	5	2	ug/L
200.7 (ICP)	Method	Water	Molybdenum	10	5	ug/L
200.7 (ICP)	Method	Water	Nickel	20	8	ug/L
200.7 (ICP)	Method	Water	Phosphorus	200	200	ug/L
200.7 (ICP)	Method	Water	Potassium	2000	900	ug/L
200.7 (ICP)	Method	Water	Selenium	100	30	ug/L
200.7 (ICP)	Method	Water	Silicon	400	20	ug/L
200.7 (ICP)	Method	Water	Silver	10	5	ug/L

METALS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
200.7 (ICP)	Method	Water	Sodium	100	70	ug/L
200.7 (ICP)	Method	Water	Strontium	10	0.4	ug/L
200.7 (ICP)	Method	Water	Thallium	200	50	ug/L
200.7 (ICP)	Method	Water	Tin	50	20	ug/L
200.7 (ICP)	Method	Water	Titanium	10	4	ug/L
200.7 (ICP)	Method	Water	Vanadium	10	6	ug/L
200.7 (ICP)	Method	Water	Zinc	10	2	ug/L
200.8	Method	Soil/Sed.	Aluminum	2	0.5	mg/kg
200.8	Method	Soil/Sed.	Antimony	0.05	0.05	mg/kg
200.8	Method	Soil/Sed.	Arsenic	0.5	0.2	mg/kg
200.8	Method	Soil/Sed.	Barium	0.05	0.02	mg/kg
200.8	Method	Soil/Sed.	Beryllium	0.02	0.005	mg/kg
200.8	Method	Soil/Sed.	Bismuth	0.1	0.006	mg/kg
200.8	Method	Soil/Sed.	Cadmium	0.02	0.009	mg/kg
200.8	Method	Soil/Sed.	Chromium	0.2	0.2	mg/kg
200.8	Method	Soil/Sed.	Cobalt	0.02	0.004	mg/kg
200.8	Method	Soil/Sed.	Copper	0.1	0.09	mg/kg
200.8	Method	Soil/Sed.	Lead	0.05	0.05	mg/kg
200.8	Method	Soil/Sed.	Manganese	0.05	0.02	mg/kg
200.8	Method	Soil/Sed.	Molybdenum	0.05	0.04	mg/kg
200.8	Method	Soil/Sed.	Nickel	0.2	0.03	mg/kg
200.8	Method	Soil/Sed.	Selenium	1	0.4	mg/kg
200.8	Method	Soil/Sed.	Silver	0.02	0.02	mg/kg
200.8	Method	Soil/Sed.	Thallium	0.02	0.005	mg/kg
200.8	Method	Soil/Sed.	Tin	0.1	0.06	mg/kg
200.8	Method	Soil/Sed.	Uranium	0.02	0.004	mg/kg
200.8	Method	Soil/Sed.	Vanadium	0.2	0.04	mg/kg
200.8	Method	Soil/Sed.	Zinc	0.5	0.5	mg/kg
200.8	Method	Water	Aluminum	2	0.5	ug/L
200.8	Method	Water	Antimony	0.05	0.02	ug/L

METALS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
200.8	Method	Water	Arsenic	0.5	0.2	ug/L
200.8	Method	Water	Barium	0.05	0.02	ug/L
200.8	Method	Water	Beryllium	0.02	0.02	ug/L
200.8	Method	Water	Bismuth	0.1	0.01	ug/L
200.8	Method	Water	Boron	0.5	0.2	ug/L
200.8	Method	Water	Cadmium	0.02	0.02	ug/L
200.8	Method	Water	Chromium	0.2	0.05	ug/L
200.8	Method	Water	Cobalt	0.02	0.008	ug/L
200.8	Method	Water	Copper	0.1	0.03	ug/L
200.8	Method	Water	Lead	0.02	0.006	ug/L
200.8	Method	Water	Manganese	0.05	0.02	ug/L
200.8	Method	Water	Molybdenum	0.05	0.04	ug/L
200.8	Method	Water	Nickel	0.2	0.05	ug/L
200.8	Method	Water	Selenium	1	0.5	ug/L
200.8	Method	Water	Silver	0.02	0.009	ug/L
200.8	Method	Water	Thallium	0.02	0.008	ug/L
200.8	Method	Water	Tin	0.1	0.03	ug/L
200.8	Method	Water	Uranium	0.02	0.02	ug/L
200.8	Method	Water	Vanadium	0.2	0.07	ug/L
200.8	Method	Water	Zinc	0.5	0.08	ug/L
200.9	Method	Soil	Arsenic	1	0.2	mg/kg
200.9	Method	Soil	Lead	1	0.09	mg/kg
200.9	Method	Soil	Selenium	1	0.06	mg/kg
200.9	Method	Soil	Thallium	1	0.2	mg/kg
200.9	Method	Water	Antimony	5	1	ug/L
200.9	Method	Water	Arsenic	5	0.7	ug/L
200.9	Method	Water	Copper	1	0.4	ug/L
200.9	Method	Water	Lead	2	1	ug/L
200.9	Method	Water	Selenium	5	0.7	ug/L
200.9	Method	Water	Thallium	5	0.4	ug/L

METALS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
6010B	3050B	Soil	Aluminum	10	9	mg/kg
6010B	3050B	Soil	Antimony	10	4	mg/kg
6010B	3050B	Soil	Arsenic	20	6	mg/kg
6010B	3050B	Soil	Barium	2	0.3	mg/kg
6010B	3050B	Soil	Beryllium	1	0.04	mg/kg
6010B	3050B	Soil	Boron	10	3	mg/kg
6010B	3050B	Soil	Cadmium	1	0.4	mg/kg
6010B	3050B	Soil	Calcium	10	4	mg/kg
6010B	3050B	Soil	Chromium	2	0.7	mg/kg
6010B	3050B	Soil	Cobalt	2	0.7	mg/kg
6010B	3050B	Soil	Copper	2	0.9	mg/kg
6010B	3050B	Soil	Iron	4	2	mg/kg
6010B	3050B	Soil	Lead	20	3	mg/kg
6010B	3050B	Soil	Lithium	2	1	mg/kg
6010B	3050B	Soil	Magnesium	4	0.8	mg/kg
6010B	3050B	Soil	Manganese	2	0.2	mg/kg
6010B	3050B	Soil	Molybdenum	2	0.7	mg/kg
6010B	3050B	Soil	Nickel	4	2	mg/kg
6010B	3050B	Soil	Phosphorus	40	30	mg/kg
6010B	3050B	Soil	Potassium	200	200	mg/kg
6010B	3050B	Soil	Selenium	40	6	mg/kg
6010B	3050B	Soil	Silver	2	0.9	mg/kg
6010B	3050B	Soil	Sodium	20	20	mg/kg
6010B	3050B	Soil	Strontium	2	0.2	mg/kg
6010B	3050B	Soil	Thallium	40	8	mg/kg
6010B	3050B	Soil	Tin	10	2	mg/kg
6010B	3050B	Soil	Titanium	2	0.5	mg/kg
6010B	3050B	Soil	Vanadium	2	0.7	mg/kg
6010B	3050B	Soil	Zinc	2	0.4	mg/kg
6010B	CLP	Water	Aluminum	50	40	ug/L

METALS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
6010B	CLP	Water	Antimony	50	20	ug/L
6010B	CLP	Water	Arsenic	100	30	ug/L
6010B	CLP	Water	Barium	5	0.9	ug/L
6010B	CLP	Water	Beryllium	5	0.2	ug/L
6010B	CLP	Water	Boron	50	8	ug/L
6010B	CLP	Water	Cadmium	5	4	ug/L
6010B	CLP	Water	Calcium	50	30	ug/L
6010B	CLP	Water	Chromium	5	3	ug/L
6010B	CLP	Water	Cobalt	10	5	ug/L
6010B	CLP	Water	Copper	10	4	ug/L
6010B	CLP	Water	Iron	20	5	ug/L
6010B	CLP	Water	Lead	50	30	ug/L
6010B	CLP	Water	Lithium	10	6	ug/L
6010B	CLP	Water	Magnesium	20	4	ug/L
6010B	CLP	Water	Manganese	5	2	ug/L
6010B	CLP	Water	Molybdenum	10	5	ug/L
6010B	CLP	Water	Nickel	20	8	ug/L
6010B	CLP	Water	Phosphorus	200	200	ug/L
6010B	CLP	Water	Potassium	2000	900	ug/L
6010B	CLP	Water	Selenium	100	30	ug/L
6010B	CLP	Water	Silicon	400	20	ug/L
6010B	CLP	Water	Silver	10	5	ug/L
6010B	CLP	Water	Sodium	100	70	ug/L
6010B	CLP	Water	Strontium	10	0.4	ug/L
6010B	CLP	Water	Thallium	200	50	ug/L
6010B	CLP	Water	Tin	50	20	ug/L
6010B	CLP	Water	Titanium	10	4	ug/L
6010B	CLP	Water	Vanadium	10	6	ug/L
6010B	CLP	Water	Zinc	10	2	ug/L
6020	3050B	Soil/Sed.	Aluminum	2	0.5	mg/kg
6020	3050B	Soil/Sed.	Antimony	0.05	0.05	mg/kg

METALS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
6020	3050B	Soil/Sed.	Arsenic	0.5	0.2	mg/kg
6020	3050B	Soil/Sed.	Barium	0.05	0.02	mg/kg
6020	3050B	Soil/Sed.	Beryllium	0.02	0.005	mg/kg
6020	3050B	Soil/Sed.	Bismuth	0.1	0.006	mg/kg
6020	3050B	Soil/Sed.	Cadmium	0.02	0.009	mg/kg
6020	3050B	Soil/Sed.	Chromium	0.2	0.2	mg/kg
6020	3050B	Soil/Sed.	Cobalt	0.02	0.004	mg/kg
6020	3050B	Soil/Sed.	Copper	0.1	0.09	mg/kg
6020	3050B	Soil/Sed.	Lead	0.05	0.05	mg/kg
6020	3050B	Soil/Sed.	Manganese	0.05	0.02	mg/kg
6020	3050B	Soil/Sed.	Molybdenum	0.05	0.04	mg/kg
6020	3050B	Soil/Sed.	Nickel	0.2	0.03	mg/kg
6020	3050B	Soil/Sed.	Selenium	1	0.4	mg/kg
6020	3050B	Soil/Sed.	Silver	0.02	0.02	mg/kg
6020	3050B	Soil/Sed.	Thallium	0.02	0.005	mg/kg
6020	3050B	Soil/Sed.	Tin	0.1	0.06	mg/kg
6020	3050B	Soil/Sed.	Uranium	0.02	0.004	mg/kg
6020	3050B	Soil/Sed.	Vanadium	0.2	0.04	mg/kg
6020	3050B	Soil/Sed.	Zinc	0.5	0.5	mg/kg
6020	CLP	Water	Aluminum	2	0.5	ug/L
6020	CLP	Water	Antimony	0.05	0.02	ug/L
6020	CLP	Water	Arsenic	0.5	0.2	ug/L
6020	CLP	Water	Barium	0.05	0.02	ug/L
6020	CLP	Water	Beryllium	0.02	0.02	ug/L
6020	CLP	Water	Bismuth	0.1	0.01	ug/L
6020	CLP	Water	Boron	0.5	0.2	ug/L
6020	CLP	Water	Cadmium	0.02	0.02	ug/L
6020	CLP	Water	Chromium	0.2	0.05	ug/L
6020	CLP	Water	Cobalt	0.02	0.008	ug/L
6020	CLP	Water	Copper	0.1	0.03	ug/L
6020	CLP	Water	Lead	0.02	0.006	ug/L

METALS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
6020	CLP	Water	Manganese	0.05	0.02	ug/L
6020	CLP	Water	Molybdenum	0.05	0.04	ug/L
6020	CLP	Water	Nickel	0.2	0.05	ug/L
6020	CLP	Water	Selenium	1	0.5	ug/L
6020	CLP	Water	Silver	0.02	0.009	ug/L
6020	CLP	Water	Thallium	0.02	0.008	ug/L
6020	CLP	Water	Tin	0.1	0.03	ug/L
6020	CLP	Water	Uranium	0.02	0.02	ug/L
6020	CLP	Water	Vanadium	0.2	0.07	ug/L
6020	CLP	Water	Zinc	0.5	0.08	ug/L
7060A	3050B	Soil	Arsenic	1	0.2	mg/kg
7062	3050B	Soil	Arsenic	0.1	0.02	mg/kg
7421	3050B	Soil	Lead	1	0.09	mg/kg
7740	3050B	Soil	Selenium	1	0.06	mg/kg
7742/*SM* 3114B	3050B	Soil	Selenium	0.1	0.02	mg/kg
7841	3050B	Soil	Thallium	1	0.2	mg/kg
7060A	CLP/3020A	Water	Arsenic	5	0.7	ug/L
7062	3010A	Water	Arsenic	1	0.2	ug/L
7191	7195	Water	Hexavalent Chromium	2	0.8	ug/L
7421	CLP/3020A	Water	Lead	2	1	ug/L
7740	CLP/3020A	Water	Selenium	5	0.7	ug/L
7742/*SM* 3114B	3010A	Water	Selenium	1	0.2	ug/L
7841	CLP/3020A	Water	Thallium	5	0.4	ug/L

a Method Detection Limits are subject to change as new MDL studies are completed.

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
1664	Method	Water	Hexane Extractable Material	5	0.9	mg/L
1664	Method	Water	Hexane Extractable Material - SGT	5	0.9	mg/L
1664	9071A	Soil	Hexane Extractable Material	100	50	mg/kg
1664	9071A	Soil	Hexane Extractable Material - SGT	100	50	mg/kg
600/4-81-045	Method	Oil	Aroclor 1016	1		mg/kg
600/4-81-045	Method	Oil	Aroclor 1221	2		mg/kg
600/4-81-045	Method	Oil	Aroclor 1232	1		mg/kg
600/4-81-045	Method	Oil	Aroclor 1242	1		mg/kg
600/4-81-045	Method	Oil	Aroclor 1248	1		mg/kg
600/4-81-045	Method	Oil	Aroclor 1254	1		mg/kg
600/4-81-045	Method	Oil	Aroclor 1260	1		mg/kg
608	3520C	Water	4,4'-DDD	0.01	0.0012	ug/L
608	3520C	Water	4,4'-DDE	0.01	0.00045	ug/L
608	3520C	Water	4,4'-DDT	0.01	0.0013	ug/L
608	3520C	Water	Aldrin	0.01	0.0012	ug/L
608	3520C	Water	alpha-BHC	0.01	0.0016	ug/L
608	3520C	Water	beta-BHC	0.01	0.015	ug/L
608	3520C	Water	Chlordane	0.2	0.11	ug/L
608	3520C	Water	delta-BHC	0.01	0.00042	ug/L
608	3520C	Water	Dieldrin	0.01	0.0013	ug/L
608	3520C	Water	Endosulfan I	0.01	0.00074	ug/L
608	3520C	Water	Endosulfan II	0.01	0.0017	ug/L
608	3520C	Water	Endosulfan Sulfate	0.01	0.0025	ug/L
608	3520C	Water	Endrin	0.01	0.00084	ug/L
608	3520C	Water	Endrin Aldehyde	0.01	0.00059	ug/L
608	3520C	Water	gamma-BHC (Lindane)	0.01	0.0011	ug/L
608	3520C	Water	Heptachlor	0.01	0.0015	ug/L
608	3520C	Water	Heptachlor Epoxide	0.01	0.00041	ug/L
608	3520C	Water	Toxaphene	0.5	0.044	ug/L
608	3520C	Water	Aroclor 1016	1	0.013	ug/L
608	3520C	Water	Aroclor 1221	1	0.16	ug/L
608	3520C	Water	Aroclor 1232	1	0.013	ug/L
608	3520C	Water	Aroclor 1242	1	0.023	ug/L
608	3520C	Water	Aroclor 1248	1	0.0068	ug/L
608	3520C	Water	Aroclor 1254	1	0.0086	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
608	3520C	Water	Aroclor 1260	1	0.0045	ug/L
625	3510C/3520C	Water	1,2,4-Trichlorobenzene	10	0.32	ug/L
625	3510C/3520C	Water	1,2-Dichlorobenzene	10	0.40	ug/L
625	3510C/3520C	Water	1,2-Diphenylhydrazine	10	0.23	ug/L
625	3510C/3520C	Water	1,3-Dichlorobenzene	10	0.37	ug/L
625	3510C/3520C	Water	1,4-Dichlorobenzene	10	0.36	ug/L
625	3510C/3520C	Water	2,3,5,6-Tetrachlorophenol	10	0.62	ug/L
625	3510C/3520C	Water	2,4,6-Trichlorophenol	10	0.19	ug/L
625	3510C/3520C	Water	2,4-Dichlorophenol	10	0.29	ug/L
625	3510C/3520C	Water	2,4-Dimethylphenol	10	1.1	ug/L
625	3510C/3520C	Water	2,4-Dinitrophenol	25	1.0	ug/L
625	3510C/3520C	Water	2,4-Dinitrotoluene	10	0.26	ug/L
625	3510C/3520C	Water	2,6-Dinitrotoluene	10	0.27	ug/L
625	3510C/3520C	Water	2-Chloronaphthalene	10	0.43	ug/L
625	3510C/3520C	Water	2-Chlorophenol	10	0.42	ug/L
625	3510C/3520C	Water	2-Methyl-4,6-dinitrophenol	25	2.3	ug/L
625	3510C/3520C	Water	2-Nitrophenol	10	0.35	ug/L
625	3510C/3520C	Water	3,3'-Dichlorobenzidine	25	0.48	ug/L
625	3510C/3520C	Water	4-Bromophenyl Phenyl Ether	10	0.35	ug/L
625	3510C/3520C	Water	4-Chloro-3-methylphenol	10	0.48	ug/L
625	3510C/3520C	Water	4-Chlorophenyl Phenyl Ether	10	0.26	ug/L
625	3510C/3520C	Water	4-Methylphenol	10	1.2	ug/L
625	3510C/3520C	Water	4-Nitrophenol	25	2.3	ug/L
625	3510C/3520C	Water	Acenaphthene	10	0.27	ug/L
625	3510C/3520C	Water	Acenaphthylene	10	0.29	ug/L
625	3510C/3520C	Water	Anthracene	10	0.33	ug/L
625	3510C/3520C	Water	Benz(a)anthracene	10	0.25	ug/L
625	3510C/3520C	Water	Benzidine	50	37	ug/L
625	3510C/3520C	Water	Benzo(a)pyrene	10	0.37	ug/L
625	3510C/3520C	Water	Benzo(b)fluoranthene	10	0.27	ug/L
625	3510C/3520C	Water	Benzo(g,h,i)perylene	10	0.41	ug/L
625	3510C/3520C	Water	Benzo(k)fluoranthene	10	0.32	ug/L
625	3510C/3520C	Water	Bis(2-chloroethoxy)methane	10	0.31	ug/L
625	3510C/3520C	Water	Bis(2-chloroethyl) Ether	10	0.37	ug/L
625	3510C/3520C	Water	Bis(2-chloroisopropyl) Ether	10	0.36	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
625	3510C/3520C	Water	Bis(2-ethylhexyl) Phthalate	10	0.34	ug/L
625	3510C/3520C	Water	Butyl Benzyl Phthalate	10	0.55	ug/L
625	3510C/3520C	Water	Chrysene	10	0.40	ug/L
625	3510C/3520C	Water	Dibenz(a,h)anthracene	10	0.41	ug/L
625	3510C/3520C	Water	Diethyl Phthalate	10	0.33	ug/L
625	3510C/3520C	Water	Dimethyl Phthalate	10	0.71	ug/L
625	3510C/3520C	Water	Di-n-butyl Phthalate	10	0.46	ug/L
625	3510C/3520C	Water	Di-n-octyl Phthalate	10	0.38	ug/L
625	3510C/3520C	Water	Fluoranthene	10	0.45	ug/L
625	3510C/3520C	Water	Fluorene	10	0.23	ug/L
625	3510C/3520C	Water	Hexachlorobenzene	10	0.27	ug/L
625	3510C/3520C	Water	Hexachlorobutadiene	10	0.22	ug/L
625	3510C/3520C	Water	Hexachlorocyclopentadiene	10	0.58	ug/L
625	3510C/3520C	Water	Hexachloroethane	10	0.26	ug/L
625	3510C/3520C	Water	Indeno(1,2,3-cd)pyrene	10	0.45	ug/L
625	3510C/3520C	Water	Isophorone	10	0.35	ug/L
625	3510C/3520C	Water	Naphthalene	10	0.31	ug/L
625	3510C/3520C	Water	Nitrobenzene	10	0.36	ug/L
625	3510C/3520C	Water	N-Nitrosodiethylamine	10	0.66	ug/L
625	3510C/3520C	Water	N-Nitrosodimethylamine	25	1.7	ug/L
625	3510C/3520C	Water	N-Nitrosodi-n-butylamine	10	0.56	ug/L
625	3510C/3520C	Water	N-Nitrosodi-n-propylamine	10	0.51	ug/L
625	3510C/3520C	Water	N-Nitrosodiphenylamine	10	0.34	ug/L
625	3510C/3520C	Water	N-Nitrosopyrrolidine	10	0.61	ug/L
625	3510C/3520C	Water	Pentachlorobenzene	10	0.54	ug/L
625	3510C/3520C	Water	Pentachlorophenol	25	0.38	ug/L
625	3510C/3520C	Water	Phenanthrene	10	0.24	ug/L
625	3510C/3520C	Water	Phenol	10	0.45	ug/L
625	3510C/3520C	Water	Pyrene	10	0.47	ug/L
1653	Method	Water	2,3,4,6-Tetrachlorophenol	2.5	0.133	ug/L
1653	Method	Water	2,4,5-Trichlorophenol	2.5	0.107	ug/L
1653	Method	Water	2,4,6-Trichlorophenol	2.5	0.212	ug/L
1653	Method	Water	3,4,5-Trichlorocatechol	5.0	0.322	ug/L
1653	Method	Water	3,4,5-Trichloroguaiacol	2.5	0.114	ug/L
1653	Method	Water	3,4,6-Trichlorocatechol	5.0	0.334	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
1653	Method	Water	3,4,6-Trichloroguaiacol	2.5	0.098	ug/L
1653	Method	Water	4,5,6-Trichloroguaiacol	2.5	0.096	ug/L
1653	Method	Water	Pentachlorophenol	5.0	0.218	ug/L
1653	Method	Water	Tetrachlorocatechol	5.0	0.184	ug/L
1653	Method	Water	Tetrachloroguaiacol	5.0	0.195	ug/L
1653	Method	Water	Trichlorosyringol	2.5	0.238	ug/L
8015B	Method	Water	Ethylene Glycol	5	1.1	mg/L
8015B	Method	Water	Propylene Glycol	5	0.77	mg/L
8015B	Method	Water	Isopropyl Ether	1	0.72	mg/L
8081A	3541	Soil	2,4'-DDD	5	0.46	ug/kg
8081A	3541	Soil	2,4'-DDE	5	0.18	ug/kg
8081A	3541	Soil	2,4'-DDT	5	0.11	ug/kg
8081A	3541	Soil	4,4'-DDD	5	0.17	ug/kg
8081A	3541	Soil	4,4'-DDE	5	0.11	ug/kg
8081A	3541	Soil	4,4'-DDT	5	0.43	ug/kg
8081A	3541	Soil	Aldrin	5	0.13	ug/kg
8081A	3541	Soil	alpha-BHC	5	0.14	ug/kg
8081A	3541	Soil	alpha-Chlordane	5	0.20	ug/kg
8081A	3541	Soil	beta-BHC	5	0.29	ug/kg
8081A	3541	Soil	Chlordane	100	11	ug/kg
8081A	3541	Soil	Chlorpyrifos	5	0.26	ug/kg
8081A	3541	Soil	cis-Nonachlor	5	0.13	ug/kg
8081A	3541	Soil	delta-BHC	5	0.18	ug/kg
8081A	3541	Soil	Dieldrin	5	0.067	ug/kg
8081A	3541	Soil	Endosulfan I	5	0.14	ug/kg
8081A	3541	Soil	Endosulfan II	5	0.50	ug/kg
8081A	3541	Soil	Endosulfan Sulfate	5	0.13	ug/kg
8081A	3541	Soil	Endrin	5	0.092	ug/kg
8081A	3541	Soil	Endrin Aldehyde	5	0.22	ug/kg
8081A	3541	Soil	Endrin Ketone	5	0.069	ug/kg
8081A	3541	Soil	gamma-BHC (Lindane)	5	0.17	ug/kg
8081A	3541	Soil	gamma-Chlordane	5	0.18	ug/kg
8081A	3541	Soil	Heptachlor	5	0.17	ug/kg
8081A	3541	Soil	Heptachlor Epoxide	5	0.15	ug/kg
8081A	3541	Soil	Hexachlorobenzene	5	0.36	ug/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8081A	3541	Soil	Hexachlorobutadiene	5	0.47	ug/kg
8081A	3541	Soil	Hexachloroethane	5	0.93	ug/kg
8081A	3541	Soil	Isodrin	5	0.17	ug/kg
8081A	3541	Soil	Methoxychlor	5	0.27	ug/kg
8081A	3541	Soil	Mirex	5	0.22	ug/kg
8081A	3541	Soil	Oxychlordane	5	0.33	ug/kg
8081A	3541	Soil	Toxaphene	250	21	ug/kg
8081A	3541	Soil	trans-Nonachlor	5	0.18	ug/kg
8081A	3540C	Soil-Low	2,4'-DDD	1.0	0.21	ug/kg
8081A	3540C	Soil-Low	2,4'-DDE	1.0	0.23	ug/kg
8081A	3540C	Soil-Low	2,4'-DDT	1.0	0.14	ug/kg
8081A	3540C	Soil-Low	4,4'-DDD	1.0	0.12	ug/kg
8081A	3540C	Soil-Low	4,4'-DDE	1.0	0.10	ug/kg
8081A	3540C	Soil-Low	4,4'-DDT	1.0	0.064	ug/kg
8081A	3540C	Soil-Low	Aldrin	1.0	0.15	ug/kg
8081A	3540C	Soil-Low	alpha-BHC	1.0	0.26	ug/kg
8081A	3540C	Soil-Low	alpha-Chlordane	1.0	0.23	ug/kg
8081A	3540C	Soil-Low	beta-BHC	1.0	0.30	ug/kg
8081A	3540C	Soil-Low	Chlordane	10	8.1	ug/kg
8081A	3540C	Soil-Low	Chlorpyrifos	1.0	0.25	ug/kg
8081A	3540C	Soil-Low	cis-Nonachlor	1.0	0.071	ug/kg
8081A	3540C	Soil-Low	delta-BHC	1.0	0.055	ug/kg
8081A	3540C	Soil-Low	Dieldrin	1.0	0.29	ug/kg
8081A	3540C	Soil-Low	Endosulfan I	1.0	0.17	ug/kg
8081A	3540C	Soil-Low	Endosulfan II	1.0	0.19	ug/kg
8081A	3540C	Soil-Low	Endosulfan Sulfate	1.0	0.079	ug/kg
8081A	3540C	Soil-Low	Endrin	1.0	0.20	ug/kg
8081A	3540C	Soil-Low	Endrin Aldehyde	1.0	0.053	ug/kg
8081A	3540C	Soil-Low	Endrin Ketone	1.0	0.082	ug/kg
8081A	3540C	Soil-Low	gamma-BHC (Lindane)	1.0	0.15	ug/kg
8081A	3540C	Soil-Low	gamma-Chlordane	1.0	0.064	ug/kg
8081A	3540C	Soil-Low	Heptachlor	1.0	0.080	ug/kg
8081A	3540C	Soil-Low	Heptachlor Epoxide	1.0	0.13	ug/kg
8081A	3540C	Soil-Low	Hexachlorobenzene	1.0	0.079	ug/kg
8081A	3540C	Soil-Low	Hexachlorobutadiene	1.0	0.13	ug/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8081A	3540C	Soil-Low	Hexachlorocyclopentadiene	1.0	0.39	ug/kg
8081A	3540C	Soil-Low	Hexachloroethane	1.0	0.15	ug/kg
8081A	3540C	Soil-Low	Isodrin	1.0	0.097	ug/kg
8081A	3540C	Soil-Low	Methoxychlor	1.0	0.10	ug/kg
8081A	3540C	Soil-Low	Mirex	1.0	0.063	ug/kg
8081A	3540C	Soil-Low	Oxychlordane	1.0	0.058	ug/kg
8081A	3540C	Soil-Low	Toxaphene	50	9.2	ug/kg
8081A	3540C	Soil-Low	trans-Nonachlor	1.0	0.066	ug/kg
8081A	3535	Water	2,4'-DDD	0.01	0.00057	ug/L
8081A	3535	Water	2,4'-DDE	0.01	0.00050	ug/L
8081A	3535	Water	2,4'-DDT	0.01	0.00059	ug/L
8081A	3535	Water	4,4'-DDD	0.01	0.00150	ug/L
8081A	3535	Water	4,4'-DDE	0.01	0.00036	ug/L
8081A	3535	Water	4,4'-DDT	0.01	0.00058	ug/L
8081A	3535	Water	Aldrin	0.01	0.00040	ug/L
8081A	3535	Water	alpha-BHC	0.01	0.00033	ug/L
8081A	3535	Water	alpha-Chlordane	0.01	0.0040	ug/L
8081A	3535	Water	beta-BHC	0.01	0.00083	ug/L
8081A	3535	Water	Chlordane	0.2	0.015	ug/L
8081A	3535	Water	Chlorpyrifos	0.01	0.00083	ug/L
8081A	3535	Water	cis-Nonachlor	0.01	0.00060	ug/L
8081A	3535	Water	delta-BHC	0.01	0.00057	ug/L
8081A	3535	Water	Dieldrin	0.01	0.00035	ug/L
8081A	3535	Water	Endosulfan I	0.01	0.00044	ug/L
8081A	3535	Water	Endosulfan II	0.01	0.00040	ug/L
8081A	3535	Water	Endosulfan Sulfate	0.01	0.00047	ug/L
8081A	3535	Water	Endrin	0.01	0.00068	ug/L
8081A	3535	Water	Endrin Aldehyde	0.01	0.00046	ug/L
8081A	3535	Water	Endrin Ketone	0.01	0.00066	ug/L
8081A	3535	Water	gamma-BHC (Lindane)	0.01	0.00044	ug/L
8081A	3535	Water	gamma-Chlordane	0.01	0.00032	ug/L
8081A	3535	Water	Heptachlor	0.01	0.00036	ug/L
8081A	3535	Water	Heptachlor Epoxide	0.01	0.00032	ug/L
8081A	3535	Water	Hexachlorobenzene	0.01	0.00031	ug/L
8081A	3535	Water	Hexachlorobutadiene	0.01	0.0019	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8081A	3535	Water	Hexachlorocyclopentadiene	0.01	0.0011	ug/L
8081A	3535	Water	Hexachloroethane	0.01	0.0012	ug/L
8081A	3535	Water	Isodrin	0.01	0.00056	ug/L
8081A	3535	Water	Methoxychlor	0.01	0.00093	ug/L
8081A	3535	Water	Mirex	0.01	0.00081	ug/L
8081A	3535	Water	Oxychlordane	0.01	0.0010	ug/L
8081A	3535	Water	Toxaphene	0.5	0.028	ug/L
8081A	3535	Water	trans-Nonachlor	0.01	0.00092	ug/L
8081A	3520C	Water-Low	2,4'-DDD	0.5	0.060	ng/L
8081A	3520C	Water-Low	2,4'-DDE	0.5	0.047	ng/L
8081A	3520C	Water-Low	2,4'-DDT	0.5	0.12	ng/L
8081A	3520C	Water-Low	4,4'-DDD	0.5	0.047	ng/L
8081A	3520C	Water-Low	4,4'-DDE	0.5	0.12	ng/L
8081A	3520C	Water-Low	4,4'-DDT	0.5	0.047	ng/L
8081A	3520C	Water-Low	Aldrin	0.5	0.14	ng/L
8081A	3520C	Water-Low	alpha-BHC	0.5	0.25	ng/L
8081A	3520C	Water-Low	alpha-Chlordane	0.5	0.044	ng/L
8081A	3520C	Water-Low	beta-BHC	0.5	0.5	ng/L
8081A	3520C	Water-Low	Chlordane	5		ng/L
8081A	3520C	Water-Low	Chlorpyrifos	0.5		ng/L
8081A	3520C	Water-Low	cis-Nonachlor	0.5		ng/L
8081A	3520C	Water-Low	delta-BHC	0.5	0.062	ng/L
8081A	3520C	Water-Low	Dieldrin	0.5	0.056	ng/L
8081A	3520C	Water-Low	Endosulfan I	0.5	0.10	ng/L
8081A	3520C	Water-Low	Endosulfan II	0.5	0.063	ng/L
8081A	3520C	Water-Low	Endosulfan Sulfate	0.5	0.13	ng/L
8081A	3520C	Water-Low	Endrin	0.5	0.054	ng/L
8081A	3520C	Water-Low	Endrin Aldehyde	0.5	0.038	ng/L
8081A	3520C	Water-Low	Endrin Ketone	0.5	0.030	ng/L
8081A	3520C	Water-Low	gamma-BHC (Lindane)	0.5	0.20	ng/L
8081A	3520C	Water-Low	gamma-Chlordane	0.5	0.065	ng/L
8081A	3520C	Water-Low	Heptachlor	0.5	0.073	ng/L
8081A	3520C	Water-Low	Heptachlor Epoxide	0.5	0.21	ng/L
8081A	3520C	Water-Low	Hexachlorobenzene	0.5	0.13	ng/L
8081A	3520C	Water-Low	Hexachlorobutadiene	0.5		ng/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8081A	3520C	Water-Low	Hexachloroethane	0.5		ng/L
8081A	3520C	Water-Low	Isodrin	0.5	0.15	ng/L
8081A	3520C	Water-Low	Methoxychlor	0.5	0.17	ng/L
8081A	3520C	Water-Low	Mirex	0.5		ng/L
8081A	3520C	Water-Low	Oxychlordane	0.5		ng/L
8081A	3520C	Water-Low	Toxaphene	25	13	ng/L
8081A	3520C	Water-Low	trans-Nonachlor	0.5		ng/L
8082 Aroclors	3540	Soil	Aroclor 1016	0.10	0.02	mg/kg
8082 Aroclors	3540	Soil	Aroclor 1221	0.20	0.02	mg/kg
8082 Aroclors	3540	Soil	Aroclor 1232	0.10	0.02	mg/kg
8082 Aroclors	3540	Soil	Aroclor 1242	0.10	0.02	mg/kg
8082 Aroclors	3540	Soil	Aroclor 1248	0.10	0.02	mg/kg
8082 Aroclors	3540	Soil	Aroclor 1254	0.10	0.02	mg/kg
8082 Aroclors	3540	Soil	Aroclor 1260	0.10	0.02	mg/kg
8082 Aroclors	3540	Soil	Aroclor 1262	0.10	0.02	mg/kg
8082 Aroclors	3540	Soil	Aroclor 1268	0.10	0.02	mg/kg
8082 Aroclors	3540C	Soil-Low	Aroclor 1016	10	1.7	ug/kg
8082 Aroclors	3540C	Soil-Low	Aroclor 1221	20	1.7	ug/kg
8082 Aroclors	3540C	Soil-Low	Aroclor 1232	10	1.7	ug/kg
8082 Aroclors	3540C	Soil-Low	Aroclor 1242	10	1.7	ug/kg
8082 Aroclors	3540C	Soil-Low	Aroclor 1248	10	1.7	ug/kg
8082 Aroclors	3540C	Soil-Low	Aroclor 1254	10	1.7	ug/kg
8082 Aroclors	3540C	Soil-Low	Aroclor 1260	10	1.7	ug/kg
8082 Aroclors	3540C	Soil-Low	Aroclor 1262	10	1.7	ug/kg
8082 Aroclors	3540C	Soil-Low	Aroclor 1268	10	1.7	ug/kg
8082 Aroclors	3535	Water	Aroclor 1016	0.2	0.0094	ug/L
8082 Aroclors	3535	Water	Aroclor 1221	0.4	0.02	ug/L
8082 Aroclors	3535	Water	Aroclor 1232	0.2	0.023	ug/L
8082 Aroclors	3535	Water	Aroclor 1242	0.2	0.013	ug/L
8082 Aroclors	3535	Water	Aroclor 1248	0.2	0.0054	ug/L
8082 Aroclors	3535	Water	Aroclor 1254	0.2	0.007	ug/L
8082 Aroclors	3535	Water	Aroclor 1260	0.2	0.0031	ug/L
8082 Aroclors	3535	Water	Aroclor 1262	0.2	0.0048	ug/L
8082 Aroclors	3535	Water	Aroclor 1268	0.2	0.0065	ug/L
8082 Aroclors	3535	Water-Low	Aroclor 1016	0.02	0.001	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8082 Aroclors	3535	Water-Low	Aroclor 1221	0.04	0.001	ug/L
8082 Aroclors	3535	Water-Low	Aroclor 1232	0.02	0.001	ug/L
8082 Aroclors	3535	Water-Low	Aroclor 1242	0.02	0.001	ug/L
8082 Aroclors	3535	Water-Low	Aroclor 1248	0.02	0.001	ug/L
8082 Aroclors	3535	Water-Low	Aroclor 1254	0.02	0.001	ug/L
8082 Aroclors	3535	Water-Low	Aroclor 1260	0.02	0.001	ug/L
8082 Aroclors	3535	Water-Low	Aroclor 1262	0.02	0.001	ug/L
8082 Aroclors	3535	Water-Low	Aroclor 1268	0.02	0.001	ug/L
8082 Aroclors	3520C	Water-UL	Aroclor 1016	0.005	0.001	ug/L
8082 Aroclors	3520C	Water-UL	Aroclor 1221	0.010	0.001	ug/L
8082 Aroclors	3520C	Water-UL	Aroclor 1232	0.005	0.001	ug/L
8082 Aroclors	3520C	Water-UL	Aroclor 1242	0.005	0.001	ug/L
8082 Aroclors	3520C	Water-UL	Aroclor 1248	0.005	0.001	ug/L
8082 Aroclors	3520C	Water-UL	Aroclor 1254	0.005	0.001	ug/L
8082 Aroclors	3520C	Water-UL	Aroclor 1260	0.005	0.001	ug/L
8082 Aroclors	3520C	Water-UL	Aroclor 1262	0.005	0.001	ug/L
8082 Aroclors	3520C	Water-UL	Aroclor 1268	0.005	0.001	ug/L
8082 Congeners	3540C	Soil	PCB1 (2-Chlorobiphenyl)	5.0		ug/kg
8082 Congeners	3540C	Soil	PCB101 (2,2',4,5,5'-Pentachlorobiphenyl)	0.50	0.090	ug/kg
8082 Congeners	3540C	Soil	PCB105 (2,3,3',4,4'-Pentachlorobiphenyl)	0.50	0.13	ug/kg
8082 Congeners	3540C	Soil	PCB110 (2,3,3',4',6-Pentachlorobiphenyl)	0.50	0.088	ug/kg
8082 Congeners	3540C	Soil	PCB114 (2,3,4,4',5-Pentachlorobiphenyl)	0.50	0.077	ug/kg
8082 Congeners	3540C	Soil	PCB118 (2,3',4,4',5-Pentachlorobiphenyl)	0.50	0.13	ug/kg
8082 Congeners	3540C	Soil	PCB119 (2,3',4,4',6-Pentachlorobiphenyl)	0.50	0.17	ug/kg
8082 Congeners	3540C	Soil	PCB123 (2',3,4,4',5-Pentachlorobiphenyl)	0.50	0.13	ug/kg
8082 Congeners	3540C	Soil	PCB126 (3,3',4,4',5-Pentachlorobiphenyl)	0.50	0.35	ug/kg
8082 Congeners	3540C	Soil	PCB128 (2,2',3,3',4,4'-Hexachlorobiphenyl)	0.50	0.14	ug/kg
8082 Congeners	3540C	Soil	PCB132 (2,2',3,3',4,6'-Hexachlorobiphenyl)	0.50	0.089	ug/kg
8082 Congeners	3540C	Soil	PCB138 (2,2',3,4,4',5'-Hexachlorobiphenyl)	0.50	0.093	ug/kg
8082 Congeners	3540C	Soil	PCB141 (2,2',3,4,5,5'-Hexachlorobiphenyl)	0.50	0.097	ug/kg
8082 Congeners	3540C	Soil	PCB149 (2,2',3,4',5',6-Hexachlorobiphenyl)	0.50	0.14	ug/kg
8082 Congeners	3540C	Soil	PCB151 (2,2',3,5,5',6-Hexachlorobiphenyl)	0.50	0.080	ug/kg
8082 Congeners	3540C	Soil	PCB153 (2,2',4,4',5,5'-Hexachlorobiphenyl)	0.50	0.18	ug/kg
8082 Congeners	3540C	Soil	PCB156 (2,3,3',4,4',5-Hexachlorobiphenyl)	0.50	0.077	ug/kg
8082 Congeners	3540C	Soil	PCB157 (2,3,3',4,4',5'-Hexachlorobiphenyl)	0.50	0.14	ug/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8082 Congeners	3540C	Soil	PCB158 (2,3,3',4,4',6-Hexachlorobiphenyl)	0.50	0.17	ug/kg
8082 Congeners	3540C	Soil	PCB166 (2,3,4,4',5,6-Hexachlorobiphenyl)	0.50	0.36	ug/kg
8082 Congeners	3540C	Soil	PCB167 (2,3',4,4',5,5'-Hexachlorobiphenyl)	0.50	0.11	ug/kg
8082 Congeners	3540C	Soil	PCB168 (2,3',4,4',5',6-Hexachlorobiphenyl)	0.50	0.14	ug/kg
8082 Congeners	3540C	Soil	PCB169 (3,3',4,4',5,5'-Hexachlorobiphenyl)	0.50	0.15	ug/kg
8082 Congeners	3540C	Soil	PCB170 (2,2',3,3',4,4',5-Heptachlorobiphenyl)	0.50	0.16	ug/kg
8082 Congeners	3540C	Soil	PCB174 (2,2',3,3',4,5,6'-Heptachlorobiphenyl)	0.50	0.11	ug/kg
8082 Congeners	3540C	Soil	PCB177 (2,2',3,3',4',5,6-Heptachlorobiphenyl)	0.50	0.13	ug/kg
8082 Congeners	3540C	Soil	PCB18 (2,2',5-Trichlorobiphenyl)	0.50	0.24	ug/kg
8082 Congeners	3540C	Soil	PCB180 (2,2',3,4,4',5,5'-Heptachlorobiphenyl)	0.50	0.076	ug/kg
8082 Congeners	3540C	Soil	PCB183 (2,2',3,4,4',5',6-Heptachlorobiphenyl)	0.50	0.078	ug/kg
8082 Congeners	3540C	Soil	PCB184 (2,2',3,4,4',6,6'-Heptachlorobiphenyl)	0.50	0.066	ug/kg
8082 Congeners	3540C	Soil	PCB187 (2,2',3,4',5,5',6-Heptachlorobiphenyl)	0.50	0.27	ug/kg
8082 Congeners	3540C	Soil	PCB189 (2,3,3',4,4',5,5'-Heptachlorobiphenyl)	0.50	0.11	ug/kg
8082 Congeners	3540C	Soil	PCB194 (2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	0.50	0.076	ug/kg
8082 Congeners	3540C	Soil	PCB195 (2,2',3,3',4,4',5,6-Octachlorobiphenyl)	0.50	0.18	ug/kg
8082 Congeners	3540C	Soil	PCB201 (2,2',3,3',4,5',6,6'-Octachlorobiphenyl)	0.50	0.10	ug/kg
8082 Congeners	3540C	Soil	PCB203 (2,2',3,4,4',5,5',6-Octachlorobiphenyl)	0.50	0.094	ug/kg
8082 Congeners	3540C	Soil	PCB206 (2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	0.50	0.094	ug/kg
8082 Congeners	3540C	Soil	PCB209 (Decachlorobiphenyl)	0.50	0.12	ug/kg
8082 Congeners	3540C	Soil	PCB28 (2,4,4'-Trichlorobiphenyl)	0.50	0.083	ug/kg
8082 Congeners	3540C	Soil	PCB31 (2,4',5-Trichlorobiphenyl)	0.50	0.51	ug/kg
8082 Congeners	3540C	Soil	PCB33 (2',3,4-Trichlorobiphenyl)	0.50	0.18	ug/kg
8082 Congeners	3540C	Soil	PCB37 (3,4,4'-Trichlorobiphenyl)	0.50	0.22	ug/kg
8082 Congeners	3540C	Soil	PCB44 (2,2',3,5'-Tetrachlorobiphenyl)	0.50	0.098	ug/kg
8082 Congeners	3540C	Soil	PCB49 (2,2',4,5'-Tetrachlorobiphenyl)	0.50	0.28	ug/kg
8082 Congeners	3540C	Soil	PCB5 (2,3-Dichlorobiphenyl)	0.50	0.12	ug/kg
8082 Congeners	3540C	Soil	PCB52 (2,2',5,5'-Tetrachlorobiphenyl)	0.50	0.088	ug/kg
8082 Congeners	3540C	Soil	PCB56 (2,3,3',4'-Tetrachlorobiphenyl)	0.50	0.077	ug/kg
8082 Congeners	3540C	Soil	PCB60 (2,3,4,4'-Tetrachlorobiphenyl)	0.50	0.14	ug/kg
8082 Congeners	3540C	Soil	PCB66 (2,3',4,4'-Tetrachlorobiphenyl)	0.50	0.070	ug/kg
8082 Congeners	3540C	Soil	PCB70 (2,3',4',5-Tetrachlorobiphenyl)	0.50	0.11	ug/kg
8082 Congeners	3540C	Soil	PCB74 (2,4,4',5-Tetrachlorobiphenyl)	0.50	0.19	ug/kg
8082 Congeners	3540C	Soil	PCB77 (3,3',4,4'-Tetrachlorobiphenyl)	0.50	0.16	ug/kg
8082 Congeners	3540C	Soil	PCB8 (2,4'-Dichlorobiphenyl)	0.50	0.19	ug/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8082 Congeners	3540C	Soil	PCB81 (3,4,4',5-Tetrachlorobiphenyl)	0.50	0.17	ug/kg
8082 Congeners	3540C	Soil	PCB87 (2,2',3,4,5'-Pentachlorobiphenyl)	0.50	0.15	ug/kg
8082 Congeners	3540C	Soil	PCB90 (2,2',3,4',5-Pentachlorobiphenyl)	0.50	0.16	ug/kg
8082 Congeners	3540C	Soil	PCB95 (2,2',3,5',6-Pentachlorobiphenyl)	0.50	0.37	ug/kg
8082 Congeners	3540C	Soil	PCB97 (2,2',3',4,5-Pentachlorobiphenyl)	0.50	0.10	ug/kg
8082 Congeners	3540C	Soil	PCB99 (2,2',4,4',5-Pentachlorobiphenyl)	0.50	0.24	ug/kg
8082 Congeners	3520C	Water	PCB1 (2-Chlorobiphenyl)	50		ng/L
8082 Congeners	3520C	Water	PCB101 (2,2',4,5,5'-Pentachlorobiphenyl)	5	0.74	ng/L
8082 Congeners	3520C	Water	PCB105 (2,3,3',4,4'-Pentachlorobiphenyl)	5	0.42	ng/L
8082 Congeners	3520C	Water	PCB110 (2,3,3',4',6-Pentachlorobiphenyl)	5	0.69	ng/L
8082 Congeners	3520C	Water	PCB114 (2,3,4,4',5-Pentachlorobiphenyl)	5	0.54	ng/L
8082 Congeners	3520C	Water	PCB118 (2,3',4,4',5-Pentachlorobiphenyl)	5	0.53	ng/L
8082 Congeners	3520C	Water	PCB119 (2,3',4,4',6-Pentachlorobiphenyl)	5	0.40	ng/L
8082 Congeners	3520C	Water	PCB123 (2',3,4,4',5-Pentachlorobiphenyl)	5	0.83	ng/L
8082 Congeners	3520C	Water	PCB126 (3,3',4,4',5-Pentachlorobiphenyl)	5	0.45	ng/L
8082 Congeners	3520C	Water	PCB128 (2,2',3,3',4,4'-Hexachlorobiphenyl)	5	0.37	ng/L
8082 Congeners	3520C	Water	PCB132 (2,2',3,3',4,6'-Hexachlorobiphenyl)	5	0.51	ng/L
8082 Congeners	3520C	Water	PCB138 (2,2',3,4,4',5'-Hexachlorobiphenyl)	5	0.65	ng/L
8082 Congeners	3520C	Water	PCB141 (2,2',3,4,5,5'-Hexachlorobiphenyl)	5	0.58	ng/L
8082 Congeners	3520C	Water	PCB149 (2,2',3,4',5',6-Hexachlorobiphenyl)	5	0.37	ng/L
8082 Congeners	3520C	Water	PCB151 (2,2',3,5,5',6-Hexachlorobiphenyl)	5	0.80	ng/L
8082 Congeners	3520C	Water	PCB153 (2,2',4,4',5,5'-Hexachlorobiphenyl)	5	0.43	ng/L
8082 Congeners	3520C	Water	PCB156 (2,3,3',4,4',5-Hexachlorobiphenyl)	5	0.52	ng/L
8082 Congeners	3520C	Water	PCB157 (2,3,3',4,4',5'-Hexachlorobiphenyl)	5	0.36	ng/L
8082 Congeners	3520C	Water	PCB158 (2,3,3',4,4',6-Hexachlorobiphenyl)	5	0.35	ng/L
8082 Congeners	3520C	Water	PCB166 (2,3,4,4',5,6-Hexachlorobiphenyl)	5	0.40	ng/L
8082 Congeners	3520C	Water	PCB167 (2,3',4,4',5,5'-Hexachlorobiphenyl)	5	0.69	ng/L
8082 Congeners	3520C	Water	PCB168 (2,3',4,4',5',6-Hexachlorobiphenyl)	5	0.40	ng/L
8082 Congeners	3520C	Water	PCB169 (3,3',4,4',5,5'-Hexachlorobiphenyl)	5	0.57	ng/L
8082 Congeners	3520C	Water	PCB170 (2,2',3,3',4,4',5-Heptachlorobiphenyl)	5	0.33	ng/L
8082 Congeners	3520C	Water	PCB174 (2,2',3,3',4,5,6'-Heptachlorobiphenyl)	5	0.45	ng/L
8082 Congeners	3520C	Water	PCB177 (2,2',3,3',4',5,6-Heptachlorobiphenyl)	5	0.74	ng/L
8082 Congeners	3520C	Water	PCB18 (2,2',5-Trichlorobiphenyl)	5	1.3	ng/L
8082 Congeners	3520C	Water	PCB180 (2,2',3,4,4',5,5'-Heptachlorobiphenyl)	5	0.64	ng/L
8082 Congeners	3520C	Water	PCB183 (2,2',3,4,4',5',6-Heptachlorobiphenyl)	5	0.56	ng/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8082 Congeners	3520C	Water	PCB184 (2,2',3,4,4',6,6'-Heptachlorobiphenyl)	5	0.73	ng/L
8082 Congeners	3520C	Water	PCB187 (2,2',3,4',5,5',6-Heptachlorobiphenyl)	5	0.90	ng/L
8082 Congeners	3520C	Water	PCB189 (2,3,3',4,4',5,5'-Heptachlorobiphenyl)	5	0.53	ng/L
8082 Congeners	3520C	Water	PCB194 (2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	5	0.49	ng/L
8082 Congeners	3520C	Water	PCB195 (2,2',3,3',4,4',5,6-Octachlorobiphenyl)	5	0.44	ng/L
8082 Congeners	3520C	Water	PCB201 (2,2',3,3',4,5',6,6'-Octachlorobiphenyl)	5	0.61	ng/L
8082 Congeners	3520C	Water	PCB203 (2,2',3,4,4',5,5',6-Octachlorobiphenyl)	5	0.49	ng/L
8082 Congeners	3520C	Water	PCB206 (2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	5	0.65	ng/L
8082 Congeners	3520C	Water	PCB209 (Decachlorobiphenyl)	5	0.64	ng/L
8082 Congeners	3520C	Water	PCB28 (2,4,4'-Trichlorobiphenyl)	5	0.48	ng/L
8082 Congeners	3520C	Water	PCB31 (2,4',5-Trichlorobiphenyl)	5	1.4	ng/L
8082 Congeners	3520C	Water	PCB33 (2',3,4-Trichlorobiphenyl)	5	0.84	ng/L
8082 Congeners	3520C	Water	PCB37 (3,4,4'-Trichlorobiphenyl)	5	0.69	ng/L
8082 Congeners	3520C	Water	PCB44 (2,2',3,5'-Tetrachlorobiphenyl)	5	1.10	ng/L
8082 Congeners	3520C	Water	PCB49 (2,2',4,5'-Tetrachlorobiphenyl)	5	0.50	ng/L
8082 Congeners	3520C	Water	PCB5 (2,3-Dichlorobiphenyl)	5	0.54	ng/L
8082 Congeners	3520C	Water	PCB52 (2,2',5,5'-Tetrachlorobiphenyl)	5	0.95	ng/L
8082 Congeners	3520C	Water	PCB56 (2,3,3',4'-Tetrachlorobiphenyl)	5	0.64	ng/L
8082 Congeners	3520C	Water	PCB60 (2,3,4,4'-Tetrachlorobiphenyl)	5	0.29	ng/L
8082 Congeners	3520C	Water	PCB66 (2,3',4,4'-Tetrachlorobiphenyl)	5	0.70	ng/L
8082 Congeners	3520C	Water	PCB70 (2,3',4',5-Tetrachlorobiphenyl)	5	0.54	ng/L
8082 Congeners	3520C	Water	PCB74 (2,4,4',5-Tetrachlorobiphenyl)	5	0.56	ng/L
8082 Congeners	3520C	Water	PCB77 (3,3',4,4'-Tetrachlorobiphenyl)	5	0.47	ng/L
8082 Congeners	3520C	Water	PCB8 (2,4'-Dichlorobiphenyl)	5	0.92	ng/L
8082 Congeners	3520C	Water	PCB81 (3,4,4',5-Tetrachlorobiphenyl)	5	0.49	ng/L
8082 Congeners	3520C	Water	PCB87 (2,2',3,4,5'-Pentachlorobiphenyl)	5	0.42	ng/L
8082 Congeners	3520C	Water	PCB90 (2,2',3,4',5-Pentachlorobiphenyl)	5	0.40	ng/L
8082 Congeners	3520C	Water	PCB95 (2,2',3,5',6-Pentachlorobiphenyl)	5	0.33	ng/L
8082 Congeners	3520C	Water	PCB97 (2,2',3',4,5-Pentachlorobiphenyl)	5	0.57	ng/L
8082 Congeners	3520C	Water	PCB99 (2,2',4,4',5-Pentachlorobiphenyl)	5	0.58	ng/L
8141A	3540	Soil	Azinphos-methyl (Guthion)	0.05	0.00047	mg/kg
8141A	3540	Soil	Bolstar (Sulprofos)	0.05	0.00048	mg/kg
8141A	3540	Soil	Chlorpyrifos	0.05	0.00062	mg/kg
8141A	3540	Soil	Coumaphos	0.1	0.00073	mg/kg
8141A	3540	Soil	Demeton-O	0.04	0.0024	mg/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8141A	3540	Soil	Demeton-O,S	1.0	0.0124	mg/kg
8141A	3540	Soil	Demeton-S	0.06	0.01	mg/kg
8141A	3540	Soil	Diazinon	0.05	0.0074	mg/kg
8141A	3540	Soil	Dichlorvos	0.05	0.0045	mg/kg
8141A	3540	Soil	Dimethoate	0.05	0.001	mg/kg
8141A	3540	Soil	Disulfoton	0.05	0.001	mg/kg
8141A	3540	Soil	EPN	0.05	0.0067	mg/kg
8141A	3540	Soil	Ethoprop (Prophos)	0.05	0.00035	mg/kg
8141A	3540	Soil	Ethyl Parathion	0.05	0.00053	mg/kg
8141A	3540	Soil	Fensulfothion	0.05	0.0011	mg/kg
8141A	3540	Soil	Fenthion	0.05	0.00057	mg/kg
8141A	3540	Soil	Malathion	0.05	0.00028	mg/kg
8141A	3540	Soil	Merphos	0.05	0.00051	mg/kg
8141A	3540	Soil	Methyl Parathion	0.05	0.00047	mg/kg
8141A	3540	Soil	Mevinphos	0.05	0.0006	mg/kg
8141A	3540	Soil	Phorate	0.05	0.0008	mg/kg
8141A	3540	Soil	Ronnel	0.05	0.00056	mg/kg
8141A	3540	Soil	Stirophos (Tetrachlorovinphos)	0.05	0.00043	mg/kg
8141A	3540	Soil	Sulfotep	0.05	0.0004	mg/kg
8141A	3540	Soil	Tokuthion (Prothiofos)	0.05	0.00067	mg/kg
8141A	3540	Soil	Trichloronate	0.05	0.0006	mg/kg
8141A	3535	Water	Azinphos-methyl (Guthion)	1.0	0.064	ug/L
8141A	3535	Water	Bolstar (Sulprofos)	0.5	0.12	ug/L
8141A	3535	Water	Chlorpyrifos	0.2	0.11	ug/L
8141A	3535	Water	Coumaphos	1.0	0.053	ug/L
8141A	3535	Water	Demeton-O	0.4	0.65	ug/L
8141A	3535	Water	Demeton-O,S	2.0	0.69	ug/L
8141A	3535	Water	Demeton-S	1.6	0.041	ug/L
8141A	3535	Water	Diazinon	0.2	0.067	ug/L
8141A	3535	Water	Dichlorvos	0.5	0.12	ug/L
8141A	3535	Water	Dimethoate	0.5	0.059	ug/L
8141A	3535	Water	Disulfoton	1.0	0.15	ug/L
8141A	3535	Water	EPN	0.2	0.078	ug/L
8141A	3535	Water	Ethoprop (Prophos)	0.2	0.16	ug/L
8141A	3535	Water	Ethyl Parathion	0.5	0.063	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8141A	3535	Water	Fensulfothion	0.5	0.13	ug/L
8141A	3535	Water	Fenthion	0.5	0.082	ug/L
8141A	3535	Water	Malathion	0.2	0.043	ug/L
8141A	3535	Water	Merphos	0.2	0.052	ug/L
8141A	3535	Water	Methyl Parathion	0.5	0.070	ug/L
8141A	3535	Water	Mevinphos	0.5	0.056	ug/L
8141A	3535	Water	Phorate	0.5	0.12	ug/L
8141A	3535	Water	Ronnel	0.2	0.088	ug/L
8141A	3535	Water	Stirophos (Tetrachlorovinphos)	0.2	0.060	ug/L
8141A	3535	Water	Sulfotep	0.2	0.075	ug/L
8141A	3535	Water	Tokuthion (Prothiofos)	0.2	0.14	ug/L
8141A	3535	Water	Trichloronate	0.2	0.11	ug/L
8151A	Method	Soil	2,4,5-T	50	4.0	ug/kg
8151A	Method	Soil	2,4,5-TP (Silvex)	50	3.6	ug/kg
8151A	Method	Soil	2,4-D	50	3.7	ug/kg
8151A	Method	Soil	2,4-DB	50	6.9	ug/kg
8151A	Method	Soil	3,5-Dichlorobenzoic Acid	50	50	ug/kg
8151A	Method	Soil	Acifluorfen	50		ug/kg
8151A	Method	Soil	Bentazon	50		ug/kg
8151A	Method	Soil	Chloramben	50		ug/kg
8151A	Method	Soil	Dacthal	50		ug/kg
8151A	Method	Soil	Dalapon	50	31	ug/kg
8151A	Method	Soil	Dicamba	50	3.5	ug/kg
8151A	Method	Soil	Dichlorprop	50	3.0	ug/kg
8151A	Method	Soil	Dinoseb	50	6.3	ug/kg
8151A	Method	Soil	MCPA	10000	500	ug/kg
8151A	Method	Soil	MCPP	10000	250	ug/kg
8151A	Method	Soil	Pentachlorophenol	50		ug/kg
8151A	Method	Soil	Picloram	50		ug/kg
8151A	Method	Water	2,4,5-T	0.2	0.042	ug/L
8151A	Method	Water	2,4,5-TP (Silvex)	0.2	0.042	ug/L
8151A	Method	Water	2,4-D	0.4	0.034	ug/L
8151A	Method	Water	2,4-DB	0.4	0.04	ug/L
8151A	Method	Water	3,5-Dichlorobenzoic Acid	0.4		ug/L
8151A	Method	Water	Acifluorfen	0.4		ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8151A	Method	Water	Bentazon	0.4		ug/L
8151A	Method	Water	Chloramben	0.4		ug/L
8151A	Method	Water	Dacthal	0.4		ug/L
8151A	Method	Water	Dalapon	0.4	0.075	ug/L
8151A	Method	Water	Dicamba	0.2	0.085	ug/L
8151A	Method	Water	Dichlorprop	0.4	0.16	ug/L
8151A	Method	Water	Dinoseb	0.2	0.045	ug/L
8151A	Method	Water	MCPA	100	8.5	ug/L
8151A	Method	Water	MCPP	100	6.3	ug/L
8151A	Method	Water	Pentachlorophenol	0.4		ug/L
8151A	Method	Water	Picloram	0.4		ug/L
8151M	Method	Soil	2,3,4,5-Tetrachlorophenol	5	0.21	ug/kg
8151M	Method	Soil	2,3,4,6-Tetrachlorophenol	5	0.3	ug/kg
8151M	Method	Soil	2,3,5,6-Tetrachlorophenol	5	0.17	ug/kg
8151M	Method	Soil	2,4,5-Trichlorophenol	5	0.55	ug/kg
8151M	Method	Soil	2,4,6-Trichlorophenol	5	0.11	ug/kg
8151M	Method	Soil	3,4,5-Trichlorophenol	10	4.3	ug/kg
8151M	Method	Soil	3,4-Dichlorophenol	10	9.4	ug/kg
8151M	Method	Soil	3,5-Dichlorophenol	10	1.7	ug/kg
8151M	Method	Soil	Pentachlorophenol	5	0.16	ug/kg
8151M	Method	Water	2,3,4,5-Tetrachlorophenol	0.5	0.22	ug/L
8151M	Method	Water	2,3,4,6-Tetrachlorophenol	0.5	0.11	ug/L
8151M	Method	Water	2,3,5,6-Tetrachlorophenol	0.5	0.076	ug/L
8151M	Method	Water	2,4,5-Trichlorophenol	1.3	1.3	ug/L
8151M	Method	Water	2,4,6-Trichlorophenol	0.5	0.15	ug/L
8151M	Method	Water	3,4,5-Trichlorophenol	1.2	1.2	ug/L
8151M	Method	Water	3,4-Dichlorophenol	2.0	0.7	ug/L
8151M	Method	Water	3,5-Dichlorophenol	2.0	0.95	ug/L
8151M	Method	Water	Pentachlorophenol	0.5	0.08	ug/L
8270C	3541	Soil	1,2,4,5-Tetrachlorobenzene	0.33	0.020	mg/kg
8270C	3541	Soil	1,2,4-Trichlorobenzene	0.33	0.0110	mg/kg
8270C	3541	Soil	1,2-Dichlorobenzene	0.33	0.0179	mg/kg
8270C	3541	Soil	1,2-Diphenylhydrazine	0.33	0.0146	mg/kg
8270C	3541	Soil	1,3,5-Trinitrobenzene	0.67	0.068	mg/kg
8270C	3541	Soil	1,3-Dichlorobenzene	0.33	0.0183	mg/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3541	Soil	1,3-Dinitrobenzene	0.33	0.073	mg/kg
8270C	3541	Soil	1,4-Dichlorobenzene	0.33	0.0175	mg/kg
8270C	3541	Soil	1,4-Dichlorobutane	0.33	0.33	mg/kg
8270C	3541	Soil	1,4-Dioxane	0.67	0.13	mg/kg
8270C	3541	Soil	1,4-Naphthoquinone	0.33	0.065	mg/kg
8270C	3541	Soil	1,6-Dinitropyrene	0.33	0.33	mg/kg
8270C	3541	Soil	1,8-Dinitropyrene	0.33	0.33	mg/kg
8270C	3541	Soil	1-Chloronaphthalene	0.33	0.33	mg/kg
8270C	3541	Soil	1-Methylnaphthalene	0.33	0.33	mg/kg
8270C	3541	Soil	1-Naphthylamine	0.33	0.036	mg/kg
8270C	3541	Soil	1-Nitropyrene	0.33	0.10	mg/kg
8270C	3541	Soil	2,3,4,6-Tetrachlorophenol	1.0	0.046	mg/kg
8270C	3541	Soil	2,4,5-Trichlorophenol	0.33	0.0171	mg/kg
8270C	3541	Soil	2,4,6-Trichlorophenol	0.33	0.0143	mg/kg
8270C	3541	Soil	2,4-Dichlorophenol	0.33	0.0164	mg/kg
8270C	3541	Soil	2,4-Dimethylphenol	0.33	0.0151	mg/kg
8270C	3541	Soil	2,4-Dinitrophenol	2.0	0.112	mg/kg
8270C	3541	Soil	2,4-Dinitrotoluene	0.33	0.0149	mg/kg
8270C	3541	Soil	2,6-Dichlorophenol	0.33	0.016	mg/kg
8270C	3541	Soil	2,6-Diisopropylnaphthalene	0.33	0.33	mg/kg
8270C	3541	Soil	2,6-Dinitrotoluene	0.33	0.0156	mg/kg
8270C	3541	Soil	2-Acetylaminofluorene	4.0	0.027	mg/kg
8270C	3541	Soil	2-Chloronaphthalene	0.33	0.0100	mg/kg
8270C	3541	Soil	2-Chlorophenol	0.33	0.0099	mg/kg
8270C	3541	Soil	2-Methyl-4,6-dinitrophenol	2.0	0.1434	mg/kg
8270C	3541	Soil	2-Methylnaphthalene	0.33	0.0110	mg/kg
8270C	3541	Soil	2-Methylphenol	0.33	0.0167	mg/kg
8270C	3541	Soil	2-Naphthylamine	0.33	0.063	mg/kg
8270C	3541	Soil	2-Nitroaniline	2.0	0.0169	mg/kg
8270C	3541	Soil	2-Nitrofluorene	2.0	0.49	mg/kg
8270C	3541	Soil	2-Nitrophenol	0.33	0.0139	mg/kg
8270C	3541	Soil	2-Picoline	0.67	0.055	mg/kg
8270C	3541	Soil	3,3'-Dichlorobenzidine	2.0	0.0270	mg/kg
8270C	3541	Soil	3,3'-Dimethylbenzidine	2.0	1.2	mg/kg
8270C	3541	Soil	3-Methylcholanthrene	0.33	0.062	mg/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3541	Soil	3-Nitroaniline	2.0	0.175	mg/kg
8270C	3541	Soil	3-Nitrophenol	0.33	0.33	mg/kg
8270C	3541	Soil	4-Aminobiphenyl	0.33	0.065	mg/kg
8270C	3541	Soil	4-Bromophenyl Phenyl Ether	0.33	0.0122	mg/kg
8270C	3541	Soil	4-Chloro-3-methylphenol	0.33	0.0166	mg/kg
8270C	3541	Soil	4-Chloroaniline	0.33	0.0144	mg/kg
8270C	3541	Soil	4-Chlorophenyl Phenyl Ether	0.33	0.0160	mg/kg
8270C	3541	Soil	4-Methylphenol	0.33	0.0168	mg/kg
8270C	3541	Soil	4-Nitroaniline	2.0	0.179	mg/kg
8270C	3541	Soil	4-Nitrophenol	2.0	0.146	mg/kg
8270C	3541	Soil	4-Nitropyrene	0.33	0.092	mg/kg
8270C	3541	Soil	4-Nitroquinoline N-Oxide	3.0	0.068	mg/kg
8270C	3541	Soil	5-Methylchrysene	0.33	0.035	mg/kg
8270C	3541	Soil	5-Nitroacenaphthene	0.33	0.028	mg/kg
8270C	3541	Soil	5-Nitro-o-toluidine	0.33	0.011	mg/kg
8270C	3541	Soil	6-Nitrochrysene	0.33	0.096	mg/kg
8270C	3541	Soil	7,12-Dimethylbenz(a)anthracene	0.33	0.015	mg/kg
8270C	3541	Soil	7H-Dibenzo(c,g)carbazole	0.33	0.049	mg/kg
8270C	3541	Soil	a,a-Dimethylphenethylamine	1.0	0.23	mg/kg
8270C	3541	Soil	Acenaphthene	0.33	0.0134	mg/kg
8270C	3541	Soil	Acenaphthylene	0.33	0.016	mg/kg
8270C	3541	Soil	Acetophenone	0.33	0.023	mg/kg
8270C	3541	Soil	Aniline	1.0	0.0216	mg/kg
8270C	3541	Soil	Anthracene	0.33	0.0139	mg/kg
8270C	3541	Soil	Aramite, Total	2.0	0.056	mg/kg
8270C	3541	Soil	Atrazine	0.33	0.017	mg/kg
8270C	3541	Soil	Azobenzene	0.33	0.0146	mg/kg
8270C	3541	Soil	Benz(a)anthracene	0.33	0.0123	mg/kg
8270C	3541	Soil	Benzaldehyde	0.33	0.021	mg/kg
8270C	3541	Soil	Benzidine	2.0	0.42	mg/kg
8270C	3541	Soil	Benzo(a)pyrene	0.33	0.0198	mg/kg
8270C	3541	Soil	Benzo(b)fluoranthene	0.33	0.0172	mg/kg
8270C	3541	Soil	Benzo(g,h,i)perylene	0.33	0.0202	mg/kg
8270C	3541	Soil	Benzo(j)fluoranthene	0.33	0.33	mg/kg
8270C	3541	Soil	Benzo(k)fluoranthene	0.33	0.0194	mg/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3541	Soil	Benzoic Acid	2.0	0.139	mg/kg
8270C	3541	Soil	Benzophenone	0.33	0.33	mg/kg
8270C	3541	Soil	Benzyl Alcohol	0.33	0.0168	mg/kg
8270C	3541	Soil	Biphenyl	0.33	0.009	mg/kg
8270C	3541	Soil	Bis(2-chloroethoxy)methane	0.33	0.0110	mg/kg
8270C	3541	Soil	Bis(2-chloroethyl) Ether	0.33	0.0117	mg/kg
8270C	3541	Soil	Bis(2-chloroisopropyl) Ether	0.33	0.0141	mg/kg
8270C	3541	Soil	Bis(2-ethylhexyl) Phthalate	0.33	0.0186	mg/kg
8270C	3541	Soil	Butyl Benzyl Phthalate	0.33	0.0163	mg/kg
8270C	3541	Soil	Caprolactam	0.67	0.147	mg/kg
8270C	3541	Soil	Carbazole	0.33	0.0112	mg/kg
8270C	3541	Soil	Chlorobenzilate	0.33	0.020	mg/kg
8270C	3541	Soil	Chrysene	0.33	0.0118	mg/kg
8270C	3541	Soil	Diallate	0.33	0.022	mg/kg
8270C	3541	Soil	Diazinon	0.33	0.33	mg/kg
8270C	3541	Soil	Dibenz(a,h)acridine	0.33	0.028	mg/kg
8270C	3541	Soil	Dibenz(a,h)anthracene	0.33	0.0275	mg/kg
8270C	3541	Soil	Dibenz(a,j)acridine	0.33	0.048	mg/kg
8270C	3541	Soil	Dibenzo(a,e)pyrene	0.67	0.17	mg/kg
8270C	3541	Soil	Dibenzo(a,h)pyrene	0.67	0.14	mg/kg
8270C	3541	Soil	Dibenzo(a,i)pyrene	0.67	0.20	mg/kg
8270C	3541	Soil	Dibenzo(a,l)pyrene	0.67	0.18	mg/kg
8270C	3541	Soil	Dibenzofuran	0.33	0.0118	mg/kg
8270C	3541	Soil	Dicyclopentadiene	0.33	0.032	mg/kg
8270C	3541	Soil	Diethyl Phthalate	0.33	0.0141	mg/kg
8270C	3541	Soil	Diethylene Glycol Dibenzoate	0.33	0.33	mg/kg
8270C	3541	Soil	Dimethoate	0.33	0.024	mg/kg
8270C	3541	Soil	Dimethyl Phthalate	0.33	0.0164	mg/kg
8270C	3541	Soil	Di-n-butyl Phthalate	0.33	0.0121	mg/kg
8270C	3541	Soil	Di-n-octyl Phthalate	0.33	0.0240	mg/kg
8270C	3541	Soil	Dinoseb	0.33	0.052	mg/kg
8270C	3541	Soil	Diphenylamine	0.33	0.010	mg/kg
8270C	3541	Soil	Disulfoton	0.33	0.016	mg/kg
8270C	3541	Soil	Ethyl Methanesulfonate	0.33	0.089	mg/kg
8270C	3541	Soil	Ethylene Glycol Butyl Ether (EGBE)	0.33	0.0058	mg/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3541	Soil	Famphur	4.0	0.071	mg/kg
8270C	3541	Soil	Fluoranthene	0.33	0.0115	mg/kg
8270C	3541	Soil	Fluorene	0.33	0.0130	mg/kg
8270C	3541	Soil	Hexachlorobenzene	0.33	0.0147	mg/kg
8270C	3541	Soil	Hexachlorobutadiene	0.33	0.0141	mg/kg
8270C	3541	Soil	Hexachlorocyclopentadiene	0.33	0.0125	mg/kg
8270C	3541	Soil	Hexachloroethane	0.33	0.0216	mg/kg
8270C	3541	Soil	Hexachlorophene	6.7	1.2	mg/kg
8270C	3541	Soil	Hexachloropropene	0.33	0.019	mg/kg
8270C	3541	Soil	Indeno(1,2,3-cd)pyrene	0.33	0.0389	mg/kg
8270C	3541	Soil	Isodrin	0.33	0.0089	mg/kg
8270C	3541	Soil	Isophorone	0.33	0.0140	mg/kg
8270C	3541	Soil	Isosafrole	0.67	0.017	mg/kg
8270C	3541	Soil	Kepone	4.0	0.39	mg/kg
8270C	3541	Soil	Malathion	0.83	0.83	mg/kg
8270C	3541	Soil	Methapyrilene	1.0	0.091	mg/kg
8270C	3541	Soil	Methyl Methanesulfonate	0.33	0.089	mg/kg
8270C	3541	Soil	Methyl Parathion	0.33	0.013	mg/kg
8270C	3541	Soil	N,N-Dimethylaniline	0.33	0.0604	mg/kg
8270C	3541	Soil	Naphthalene	0.33	0.0144	mg/kg
8270C	3541	Soil	n-Dodecane	0.33	0.33	mg/kg
8270C	3541	Soil	Nitrobenzene	0.33	0.0261	mg/kg
8270C	3541	Soil	N-Nitrosodiethylamine	0.33	0.029	mg/kg
8270C	3541	Soil	N-Nitrosodimethylamine	2.0	0.0251	mg/kg
8270C	3541	Soil	N-Nitrosodi-n-butylamine	0.33	0.11	mg/kg
8270C	3541	Soil	N-Nitrosodi-n-propylamine	0.33	0.0191	mg/kg
8270C	3541	Soil	N-Nitrosodiphenylamine	0.33	0.018	mg/kg
8270C	3541	Soil	N-Nitrosomethylethylamine	0.33	0.099	mg/kg
8270C	3541	Soil	N-Nitrosomorpholine	0.33	0.019	mg/kg
8270C	3541	Soil	N-Nitrosopiperidine	0.33	0.023	mg/kg
8270C	3541	Soil	N-Nitrosopyrrolidine	0.33	0.092	mg/kg
8270C	3541	Soil	O,O,O-Triethyl Phosphorothioate	0.33	0.022	mg/kg
8270C	3541	Soil	o-Toluidine	0.33	0.017	mg/kg
8270C	3541	Soil	Parathion	0.33	0.084	mg/kg
8270C	3541	Soil	p-Dimethylaminoazobenzene	0.33	0.012	mg/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3541	Soil	Pentachlorobenzene	0.33	0.019	mg/kg
8270C	3541	Soil	Pentachloroethane	1.0	0.015	mg/kg
8270C	3541	Soil	Pentachloronitrobenzene	2.0	0.088	mg/kg
8270C	3541	Soil	Pentachlorophenol	2.0	0.125	mg/kg
8270C	3541	Soil	Phenacetin	2.0	0.013	mg/kg
8270C	3541	Soil	Phenanthrene	0.33	0.0100	mg/kg
8270C	3541	Soil	Phenol	0.33	0.0195	mg/kg
8270C	3541	Soil	Phorate	0.33	0.026	mg/kg
8270C	3541	Soil	Picric Acid	3.3	3.3	mg/kg
8270C	3541	Soil	p-Phenylenediamine	2.0	0.79	mg/kg
8270C	3541	Soil	Pronamide	0.33	0.018	mg/kg
8270C	3541	Soil	Pyrene	0.33	0.0140	mg/kg
8270C	3541	Soil	Pyridine	0.33	0.0199	mg/kg
8270C	3541	Soil	Safrole	0.33	0.013	mg/kg
8270C	3541	Soil	Sulfotep	0.33	0.018	mg/kg
8270C	3541	Soil	Thionazin	2.0	0.017	mg/kg
8270C	3541	Soil-Low	1,2,4,5-Tetrachlorobenzene	10	4.9	ug/kg
8270C	3541	Soil-Low	1,2,4-Trichlorobenzene	10	2.6	ug/kg
8270C	3541	Soil-Low	1,2-Dichlorobenzene	10	2.9	ug/kg
8270C	3541	Soil-Low	1,2-Diphenylhydrazine	10	2.4	ug/kg
8270C	3541	Soil-Low	1,3-Dichlorobenzene	10	3.0	ug/kg
8270C	3541	Soil-Low	1,4-Dichlorobenzene	10	2.9	ug/kg
8270C	3541	Soil-Low	2,4,5-Trichlorophenol	10	1.5	ug/kg
8270C	3541	Soil-Low	2,4,6-Trichlorophenol	10	1.4	ug/kg
8270C	3541	Soil-Low	2,4-Dichlorophenol	10	1.0	ug/kg
8270C	3541	Soil-Low	2,4-Dimethylphenol	50	5.5	ug/kg
8270C	3541	Soil-Low	2,4-Dinitrophenol	200	17	ug/kg
8270C	3541	Soil-Low	2,4-Dinitrotoluene	10	1.5	ug/kg
8270C	3541	Soil-Low	2,6-Dinitrotoluene	10	2.0	ug/kg
8270C	3541	Soil-Low	2-Chloronaphthalene	10	1.6	ug/kg
8270C	3541	Soil-Low	2-Chlorophenol	10	2.0	ug/kg
8270C	3541	Soil-Low	2-Methyl-4,6-dinitrophenol	100	1.4	ug/kg
8270C	3541	Soil-Low	2-Methylnaphthalene	10	2.2	ug/kg
8270C	3541	Soil-Low	2-Methylphenol	10	1.5	ug/kg
8270C	3541	Soil-Low	2-Nitroaniline	20	3.2	ug/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3541	Soil-Low	2-Nitrophenol	10	1.5	ug/kg
8270C	3541	Soil-Low	3,3'-Dichlorobenzidine	100	3.7	ug/kg
8270C	3541	Soil-Low	3-Nitroaniline	20	2.5	ug/kg
8270C	3541	Soil-Low	4-Bromophenyl Phenyl Ether	10	1.6	ug/kg
8270C	3541	Soil-Low	4-Chloro-3-methylphenol	10	1.4	ug/kg
8270C	3541	Soil-Low	4-Chloroaniline	10	1.9	ug/kg
8270C	3541	Soil-Low	4-Chlorophenyl Phenyl Ether	10	1.4	ug/kg
8270C	3541	Soil-Low	4-Methylphenol	10	1.5	ug/kg
8270C	3541	Soil-Low	4-Nitroaniline	20	1.8	ug/kg
8270C	3541	Soil-Low	4-Nitrophenol	100	18	ug/kg
8270C	3541	Soil-Low	Acenaphthene	10	1.4	ug/kg
8270C	3541	Soil-Low	Acenaphthylene	10	1.2	ug/kg
8270C	3541	Soil-Low	Acetophenone	50	12	ug/kg
8270C	3541	Soil-Low	Aniline	20	1.5	ug/kg
8270C	3541	Soil-Low	Anthracene	10	1.6	ug/kg
8270C	3541	Soil-Low	Atrazine	10	3.8	ug/kg
8270C	3541	Soil-Low	Azobenzene	10	1.1	ug/kg
8270C	3541	Soil-Low	Benz(a)anthracene	10	1.7	ug/kg
8270C	3541	Soil-Low	Benzaldehyde	20	7.7	ug/kg
8270C	3541	Soil-Low	Benzidine	200	200	ug/kg
8270C	3541	Soil-Low	Benzo(a)pyrene	10	1.7	ug/kg
8270C	3541	Soil-Low	Benzo(b)fluoranthene	10	1.2	ug/kg
8270C	3541	Soil-Low	Benzo(g,h,i)perylene	10	1.5	ug/kg
8270C	3541	Soil-Low	Benzo(k)fluoranthene	10	1.4	ug/kg
8270C	3541	Soil-Low	Benzoic Acid	200	96	ug/kg
8270C	3541	Soil-Low	Benzyl Alcohol	20	2.1	ug/kg
8270C	3541	Soil-Low	Biphenyl	20	1.9	ug/kg
8270C	3541	Soil-Low	Bis(2-chloroethoxy)methane	10	1.5	ug/kg
8270C	3541	Soil-Low	Bis(2-chloroethyl) Ether	10	1.9	ug/kg
8270C	3541	Soil-Low	Bis(2-chloroisopropyl) Ether	10	2.6	ug/kg
8270C	3541	Soil-Low	Bis(2-ethylhexyl) Phthalate	100	7.0	ug/kg
8270C	3541	Soil-Low	Butyl Benzyl Phthalate	10	3.2	ug/kg
8270C	3541	Soil-Low	Caprolactam	50	19	ug/kg
8270C	3541	Soil-Low	Carbazole	10	1.3	ug/kg
8270C	3541	Soil-Low	Chrysene	10	1.5	ug/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3541	Soil-Low	Dibenz(a,h)anthracene	10	1.5	ug/kg
8270C	3541	Soil-Low	Dibenzofuran	10	1.2	ug/kg
8270C	3541	Soil-Low	Diethyl Phthalate	10	1.3	ug/kg
8270C	3541	Soil-Low	Dimethyl Phthalate	10	1.0	ug/kg
8270C	3541	Soil-Low	Di-n-butyl Phthalate	20	7.9	ug/kg
8270C	3541	Soil-Low	Di-n-octyl Phthalate	10	1.7	ug/kg
8270C	3541	Soil-Low	Fluoranthene	10	1.6	ug/kg
8270C	3541	Soil-Low	Fluorene	10	1.1	ug/kg
8270C	3541	Soil-Low	Hexachlorobenzene	10	1.2	ug/kg
8270C	3541	Soil-Low	Hexachlorobutadiene	10	2.5	ug/kg
8270C	3541	Soil-Low	Hexachlorocyclopentadiene	50	29	ug/kg
8270C	3541	Soil-Low	Hexachloroethane	10	3.1	ug/kg
8270C	3541	Soil-Low	HPAH	10	10	ug/kg
8270C	3541	Soil-Low	Indeno(1,2,3-cd)pyrene	10	1.5	ug/kg
8270C	3541	Soil-Low	Isophorone	10	1.0	ug/kg
8270C	3541	Soil-Low	LPAH	10	10	ug/kg
8270C	3541	Soil-Low	Naphthalene	10	2.3	ug/kg
8270C	3541	Soil-Low	Nitrobenzene	10	2.2	ug/kg
8270C	3541	Soil-Low	N-Nitrosodimethylamine	50	6.1	ug/kg
8270C	3541	Soil-Low	N-Nitrosodi-n-propylamine	10	2.4	ug/kg
8270C	3541	Soil-Low	N-Nitrosodiphenylamine	10	1.6	ug/kg
8270C	3541	Soil-Low	Pentachlorophenol	100	20	ug/kg
8270C	3541	Soil-Low	Phenanthrene	10	1.4	ug/kg
8270C	3541	Soil-Low	Phenol	30	2.0	ug/kg
8270C	3541	Soil-Low	Pyrene	10	1.5	ug/kg
8270C	3541	Soil-Low	Pyridine	50	50	ug/kg
8270C	3520C	Water	1,2,4,5-Tetrachlorobenzene	10	0.26	ug/L
8270C	3520C	Water	1,2,4-Trichlorobenzene	10	0.355	ug/L
8270C	3520C	Water	1,2-Dichlorobenzene	10	0.431	ug/L
8270C	3520C	Water	1,2-Diphenylhydrazine	10	0.509	ug/L
8270C	3520C	Water	1,3,5-Trinitrobenzene	25	0.38	ug/L
8270C	3520C	Water	1,3-Dichlorobenzene	10	0.352	ug/L
8270C	3520C	Water	1,3-Dinitrobenzene	10	0.52	ug/L
8270C	3520C	Water	1,4-Dichlorobenzene	10	0.317	ug/L
8270C	3520C	Water	1,4-Dichlorobutane	10	1	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3520C	Water	1,4-Dioxane	25	3.7	ug/L
8270C	3520C	Water	1,4-Naphthoquinone	10	0.21	ug/L
8270C	3520C	Water	1,6-Dinitropyrene	10	10	ug/L
8270C	3520C	Water	1,8-Dinitropyrene	10	10	ug/L
8270C	3520C	Water	1-Chloronaphthalene	10	1	ug/L
8270C	3520C	Water	1-Methylnaphthalene	10	10	ug/L
8270C	3520C	Water	1-Naphthylamine	10	0.70	ug/L
8270C	3520C	Water	1-Nitropyrene	10	0.53	ug/L
8270C	3520C	Water	2,3,4,6-Tetrachlorophenol	10	0.55	ug/L
8270C	3520C	Water	2,3,5,6-Tetrachlorophenol	10	0.62	ug/L
8270C	3520C	Water	2,4,5-Trichlorophenol	10	0.381	ug/L
8270C	3520C	Water	2,4,6-Trichlorophenol	10	0.203	ug/L
8270C	3520C	Water	2,4-Dichlorophenol	10	0.297	ug/L
8270C	3520C	Water	2,4-Dimethylphenol	10	0.264	ug/L
8270C	3520C	Water	2,4-Dinitrophenol	25	2.22	ug/L
8270C	3520C	Water	2,4-Dinitrotoluene	10	0.274	ug/L
8270C	3520C	Water	2,6-Dichlorophenol	10	0.48	ug/L
8270C	3520C	Water	2,6-Dinitrotoluene	10	0.349	ug/L
8270C	3520C	Water	2-Acetylaminofluorene	100	0.23	ug/L
8270C	3520C	Water	2-Chloronaphthalene	10	0.290	ug/L
8270C	3520C	Water	2-Chlorophenol	10	0.311	ug/L
8270C	3520C	Water	2-Methyl-4,6-dinitrophenol	25	2.12	ug/L
8270C	3520C	Water	2-Methylnaphthalene	10	0.239	ug/L
8270C	3520C	Water	2-Methylphenol	10	0.328	ug/L
8270C	3520C	Water	2-Naphthylamine	10	1.0	ug/L
8270C	3520C	Water	2-Nitroaniline	25	0.336	ug/L
8270C	3520C	Water	2-Nitrofluorene	50	15	ug/L
8270C	3520C	Water	2-Nitrophenol	10	0.373	ug/L
8270C	3520C	Water	2-Picoline	25	4.5	ug/L
8270C	3520C	Water	3- and 4-Methylphenol Coelution	10	0.478	ug/L
8270C	3520C	Water	3,3'-Dichlorobenzidine	25	0.270	ug/L
8270C	3520C	Water	3,3'-Dimethylbenzidine	20	5.4	ug/L
8270C	3520C	Water	3-Methylcholanthrene	10	0.31	ug/L
8270C	3520C	Water	3-Nitroaniline	25	3.25	ug/L
8270C	3520C	Water	3-Nitrophenol	10	1	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3520C	Water	4-Aminobiphenyl	10	1.4	ug/L
8270C	3520C	Water	4-Bromophenyl Phenyl Ether	10	0.274	ug/L
8270C	3520C	Water	4-Chloro-3-methylphenol	10	0.490	ug/L
8270C	3520C	Water	4-Chloroaniline	10	0.375	ug/L
8270C	3520C	Water	4-Chlorophenyl Phenyl Ether	10	0.278	ug/L
8270C	3520C	Water	4-Methylphenol	10	0.478	ug/L
8270C	3520C	Water	4-Nitroaniline	25	4.03	ug/L
8270C	3520C	Water	4-Nitrophenol	25	1.92	ug/L
8270C	3520C	Water	4-Nitropyrene	10	2.9	ug/L
8270C	3520C	Water	4-Nitroquinoline N-Oxide	100	4.6	ug/L
8270C	3520C	Water	5-Methylchrysene	10	1.1	ug/L
8270C	3520C	Water	5-Nitroacenaphthene	10	0.78	ug/L
8270C	3520C	Water	5-Nitro-o-toluidine	10	1.0	ug/L
8270C	3520C	Water	6-Nitrochrysene	10	2.4	ug/L
8270C	3520C	Water	7,12-Dimethylbenz(a)anthracene	10	0.32	ug/L
8270C	3520C	Water	7H-Dibenzo(c,g)carbazole	10	0.90	ug/L
8270C	3520C	Water	a,a-Dimethylphenethylamine	25	6.4	ug/L
8270C	3520C	Water	Acenaphthene	10	0.281	ug/L
8270C	3520C	Water	Acenaphthylene	10	0.236	ug/L
8270C	3520C	Water	Acetophenone	10	0.60	ug/L
8270C	3520C	Water	alpha-Terpineol	10	10	ug/L
8270C	3520C	Water	Aniline	25	0.487	ug/L
8270C	3520C	Water	Anthracene	10	0.612	ug/L
8270C	3520C	Water	Aramite, Total	50	0.52	ug/L
8270C	3520C	Water	Atrazine	10	0.46	ug/L
8270C	3520C	Water	Azobenzene	10	0.509	ug/L
8270C	3520C	Water	Benz(a)anthracene	10	0.591	ug/L
8270C	3520C	Water	Benzaldehyde	10	0.50	ug/L
8270C	3520C	Water	Benzidine	50	19	ug/L
8270C	3520C	Water	Benzo(a)pyrene	10	0.651	ug/L
8270C	3520C	Water	Benzo(b)fluoranthene	10	0.584	ug/L
8270C	3520C	Water	Benzo(g,h,i)perylene	10	0.812	ug/L
8270C	3520C	Water	Benzo(j)fluoranthene	10	10	ug/L
8270C	3520C	Water	Benzo(k)fluoranthene	10	0.827	ug/L
8270C	3520C	Water	Benzoic acid	25	5.819	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3520C	Water	Benzyl alcohol	10	0.377	ug/L
8270C	3520C	Water	Biphenyl	10	0.66	ug/L
8270C	3520C	Water	Bis(2-chloroethoxy)methane	10	0.276	ug/L
8270C	3520C	Water	Bis(2-chloroethyl) Ether	10	0.333	ug/L
8270C	3520C	Water	Bis(2-chloroisopropyl) Ether	10	0.311	ug/L
8270C	3520C	Water	Bis(2-ethylhexyl) Phthalate	10	1.89	ug/L
8270C	3520C	Water	Butyl Benzyl Phthalate	10	0.470	ug/L
8270C	3520C	Water	Caprolactam	25	0.58	ug/L
8270C	3520C	Water	Carbazole	10	0.237	ug/L
8270C	3520C	Water	Chlorobenzilate	10	0.45	ug/L
8270C	3520C	Water	Chrysene	10	0.787	ug/L
8270C	3520C	Water	Diallate	10	0.47	ug/L
8270C	3520C	Water	Dibenz(a,h)acridine	10	0.90	ug/L
8270C	3520C	Water	Dibenz(a,h)anthracene	10	0.752	ug/L
8270C	3520C	Water	Dibenz(a,j)acridine	10	6.1	ug/L
8270C	3520C	Water	Dibenzo(a,e)pyrene	10	2.8	ug/L
8270C	3520C	Water	Dibenzo(a,h)pyrene	10	5.9	ug/L
8270C	3520C	Water	Dibenzo(a,i)pyrene	10	2.6	ug/L
8270C	3520C	Water	Dibenzo(a,l)pyrene	10	1.2	ug/L
8270C	3520C	Water	Dibenzofuran	10	0.325	ug/L
8270C	3520C	Water	Dicyclopentadiene	10	0.59	ug/L
8270C	3520C	Water	Diethyl Phthalate	10	0.289	ug/L
8270C	3520C	Water	Dimethoate	10	0.69	ug/L
8270C	3520C	Water	Dimethyl Phthalate	10	0.254	ug/L
8270C	3520C	Water	Di-n-butyl Phthalate	10	0.364	ug/L
8270C	3520C	Water	Di-n-octyl Phthalate	10	0.626	ug/L
8270C	3520C	Water	Dinoseb	10	0.42	ug/L
8270C	3520C	Water	Diphenylamine	10	0.42	ug/L
8270C	3520C	Water	Disulfoton	10	0.57	ug/L
8270C	3520C	Water	Ethyl Methanesulfonate	10	0.28	ug/L
8270C	3520C	Water	Famphur	10	0.27	ug/L
8270C	3520C	Water	Fluoranthene	10	0.652	ug/L
8270C	3520C	Water	Fluorene	10	0.323	ug/L
8270C	3520C	Water	Hexachlorobenzene	10	0.628	ug/L
8270C	3520C	Water	Hexachlorobutadiene	10	0.291	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3520C	Water	Hexachlorocyclopentadiene	10	1.21	ug/L
8270C	3520C	Water	Hexachloroethane	10	0.289	ug/L
8270C	3520C	Water	Hexachlorophene	150	44	ug/L
8270C	3520C	Water	Hexachloropropene	10	0.19	ug/L
8270C	3520C	Water	Indeno(1,2,3-cd)pyrene	10	0.684	ug/L
8270C	3520C	Water	Isodrin	10	0.36	ug/L
8270C	3520C	Water	Isophorone	10	0.246	ug/L
8270C	3520C	Water	Isosafrole	10	0.48	ug/L
8270C	3520C	Water	Kepone	20	4.1	ug/L
8270C	3520C	Water	Malathion	25	1	ug/L
8270C	3520C	Water	Methapyrilene	100	9.3	ug/L
8270C	3520C	Water	Methyl Methanesulfonate	10	0.31	ug/L
8270C	3520C	Water	Methyl Parathion	10	0.51	ug/L
8270C	3520C	Water	N,N-Dimethylaniline	10	2.22	ug/L
8270C	3520C	Water	Naphthalene	10	0.365	ug/L
8270C	3520C	Water	Nitrobenzene	10	0.567	ug/L
8270C	3520C	Water	N-Nitrosodiethylamine	10	0.41	ug/L
8270C	3520C	Water	N-Nitrosodimethylamine	25	0.479	ug/L
8270C	3520C	Water	N-Nitrosodi-n-butylamine	10	0.57	ug/L
8270C	3520C	Water	N-Nitrosodi-n-propylamine	10	0.496	ug/L
8270C	3520C	Water	N-Nitrosodiphenylamine	10	0.48	ug/L
8270C	3520C	Water	N-Nitrosomethylethylamine	25	4.6	ug/L
8270C	3520C	Water	N-Nitrosomorpholine	10	0.25	ug/L
8270C	3520C	Water	N-Nitrosopiperidine	10	0.32	ug/L
8270C	3520C	Water	N-Nitrosopyrrolidine	10	0.39	ug/L
8270C	3520C	Water	O,O,O-Triethyl Phosphorothioate	10	0.37	ug/L
8270C	3520C	Water	o-Toluidine	10	1.4	ug/L
8270C	3520C	Water	Parathion	10	0.51	ug/L
8270C	3520C	Water	p-Dimethylaminoazobenzene	10	0.31	ug/L
8270C	3520C	Water	Pentachlorobenzene	10	0.31	ug/L
8270C	3520C	Water	Pentachloroethane	25	0.28	ug/L
8270C	3520C	Water	Pentachloronitrobenzene	50	0.23	ug/L
8270C	3520C	Water	Pentachlorophenol	25	2.44	ug/L
8270C	3520C	Water	Phenacetin	50	0.42	ug/L
8270C	3520C	Water	Phenanthrene	10	0.482	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3520C	Water	Phenol	10	0.324	ug/L
8270C	3520C	Water	Phorate	10	0.36	ug/L
8270C	3520C	Water	p-Phenylenediamine	100	19	ug/L
8270C	3520C	Water	Pronamide	10	0.41	ug/L
8270C	3520C	Water	Pyrene	10	0.731	ug/L
8270C	3520C	Water	Pyridine	25	7.50	ug/L
8270C	3520C	Water	Quinoline	10	10	ug/L
8270C	3520C	Water	Safrole	10	0.36	ug/L
8270C	3520C	Water	Sulfotep	10	0.26	ug/L
8270C	3520C	Water	Thionazin	25	0.71	ug/L
8270C	3520C	Water-Low	1,2,4,5-Tetrachlorobenzene	0.2	0.057	ug/L
8270C	3520C	Water-Low	1,2,4-Trichlorobenzene	0.2	0.016	ug/L
8270C	3520C	Water-Low	1,2-Dichlorobenzene	0.2	0.022	ug/L
8270C	3520C	Water-Low	1,2-Diphenylhydrazine	0.2	0.021	ug/L
8270C	3520C	Water-Low	1,3-Dichlorobenzene	0.2	0.021	ug/L
8270C	3520C	Water-Low	1,4-Dichlorobenzene	0.2	0.029	ug/L
8270C	3520C	Water-Low	2,4,5-Trichlorophenol	0.5	0.031	ug/L
8270C	3520C	Water-Low	2,4,6-Trichlorophenol	0.5	0.058	ug/L
8270C	3520C	Water-Low	2,4-Dichlorophenol	0.5	0.047	ug/L
8270C	3520C	Water-Low	2,4-Dimethylphenol	4.0	2.2	ug/L
8270C	3520C	Water-Low	2,4-Dinitrophenol	4.0	0.17	ug/L
8270C	3520C	Water-Low	2,4-Dinitrotoluene	0.2	0.018	ug/L
8270C	3520C	Water-Low	2,6-Dinitrotoluene	0.2	0.033	ug/L
8270C	3520C	Water-Low	2-Chloronaphthalene	0.2	0.041	ug/L
8270C	3520C	Water-Low	2-Chlorophenol	0.5	0.054	ug/L
8270C	3520C	Water-Low	2-Methyl-4,6-dinitrophenol	2.0	0.025	ug/L
8270C	3520C	Water-Low	2-Methylnaphthalene	0.2	0.026	ug/L
8270C	3520C	Water-Low	2-Methylphenol	0.5	0.11	ug/L
8270C	3520C	Water-Low	2-Nitroaniline	0.2	0.024	ug/L
8270C	3520C	Water-Low	2-Nitrophenol	0.5	0.063	ug/L
8270C	3520C	Water-Low	3,3'-Dichlorobenzidine	2.0	0.428	ug/L
8270C	3520C	Water-Low	3-Nitroaniline	1.0	0.029	ug/L
8270C	3520C	Water-Low	4-Bromophenyl Phenyl Ether	0.2	0.026	ug/L
8270C	3520C	Water-Low	4-Chloro-3-methylphenol	0.5	0.037	ug/L
8270C	3520C	Water-Low	4-Chloroaniline	0.2	0.025	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3520C	Water-Low	4-Chlorophenyl Phenyl Ether	0.2	0.027	ug/L
8270C	3520C	Water-Low	4-Methylphenol	0.5	0.12	ug/L
8270C	3520C	Water-Low	4-Nitroaniline	1.0	0.019	ug/L
8270C	3520C	Water-Low	4-Nitrophenol	2.0	0.28	ug/L
8270C	3520C	Water-Low	Acenaphthene	0.2	0.026	ug/L
8270C	3520C	Water-Low	Acenaphthylene	0.2	0.015	ug/L
8270C	3520C	Water-Low	Acetophenone	0.5	0.16	ug/L
8270C	3520C	Water-Low	Aniline	1.0	0.043	ug/L
8270C	3520C	Water-Low	Anthracene	0.2	0.024	ug/L
8270C	3520C	Water-Low	Atrazine	0.2	0.053	ug/L
8270C	3520C	Water-Low	Azobenzene	0.2	0.021	ug/L
8270C	3520C	Water-Low	Benz(a)anthracene	0.2	0.018	ug/L
8270C	3520C	Water-Low	Benzaldehyde	0.2	0.046	ug/L
8270C	3520C	Water-Low	Benzo(a)pyrene	0.2	0.031	ug/L
8270C	3520C	Water-Low	Benzo(b)fluoranthene	0.2	0.017	ug/L
8270C	3520C	Water-Low	Benzo(g,h,i)perylene	0.2	0.019	ug/L
8270C	3520C	Water-Low	Benzo(k)fluoranthene	0.2	0.024	ug/L
8270C	3520C	Water-Low	Benzoic Acid	5.0	1.1	ug/L
8270C	3520C	Water-Low	Benzyl Alcohol	5.0	0.073	ug/L
8270C	3520C	Water-Low	Biphenyl	0.2	0.037	ug/L
8270C	3520C	Water-Low	Bis(2-chloroethoxy)methane	0.2	0.024	ug/L
8270C	3520C	Water-Low	Bis(2-chloroethyl) Ether	0.2	0.035	ug/L
8270C	3520C	Water-Low	Bis(2-chloroisopropyl) Ether	0.2	0.026	ug/L
8270C	3520C	Water-Low	Bis(2-ethylhexyl) Phthalate	1.0	0.13	ug/L
8270C	3520C	Water-Low	Butyl Benzyl Phthalate	0.2	0.018	ug/L
8270C	3520C	Water-Low	Caprolactam	0.50	0.22	ug/L
8270C	3520C	Water-Low	Carbazole	0.2	0.018	ug/L
8270C	3520C	Water-Low	Chrysene	0.2	0.028	ug/L
8270C	3520C	Water-Low	Dibenz(a,h)anthracene	0.2	0.017	ug/L
8270C	3520C	Water-Low	Dibenzofuran	0.2	0.018	ug/L
8270C	3520C	Water-Low	Diethyl Phthalate	0.2	0.012	ug/L
8270C	3520C	Water-Low	Dimethyl Phthalate	0.2	0.021	ug/L
8270C	3520C	Water-Low	Di-n-butyl Phthalate	0.2	0.023	ug/L
8270C	3520C	Water-Low	Di-n-octyl Phthalate	0.2	0.018	ug/L
8270C	3520C	Water-Low	Fluoranthene	0.2	0.020	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3520C	Water-Low	Fluorene	0.2	0.027	ug/L
8270C	3520C	Water-Low	Hexachlorobenzene	0.2	0.022	ug/L
8270C	3520C	Water-Low	Hexachlorobutadiene	0.2	0.027	ug/L
8270C	3520C	Water-Low	Hexachlorocyclopentadiene	1.0	0.19	ug/L
8270C	3520C	Water-Low	Hexachloroethane	0.2	0.024	ug/L
8270C	3520C	Water-Low	Indeno(1,2,3-cd)pyrene	0.2	0.021	ug/L
8270C	3520C	Water-Low	Isophorone	0.2	0.016	ug/L
8270C	3520C	Water-Low	Naphthalene	0.2	0.022	ug/L
8270C	3520C	Water-Low	Nitrobenzene	0.2	0.028	ug/L
8270C	3520C	Water-Low	N-Nitrosodimethylamine	2.0	0.42	ug/L
8270C	3520C	Water-Low	N-Nitrosodi-n-propylamine	0.2	0.037	ug/L
8270C	3520C	Water-Low	N-Nitrosodiphenylamine	0.2	0.048	ug/L
8270C	3520C	Water-Low	Pentachlorophenol	1.0	0.34	ug/L
8270C	3520C	Water-Low	Phenanthrene	0.2	0.022	ug/L
8270C	3520C	Water-Low	Phenol	0.5	0.063	ug/L
8270C	3520C	Water-Low	Pyrene	0.2	0.019	ug/L
8270C	3520C	Water-Low	Pyridine	5.0	1.4	ug/L
8270C-SIM PAH	3541	Soil	1-Methylnaphthalene	5	0.31	ug/kg
8270C-SIM PAH	3541	Soil	1-Methylphenanthrene	5	0.28	ug/kg
8270C-SIM PAH	3541	Soil	2,3,5-Trimethylnaphthalene	5	0.21	ug/kg
8270C-SIM PAH	3541	Soil	2,6-Dimethylnaphthalene	5	0.36	ug/kg
8270C-SIM PAH	3541	Soil	2-Methylnaphthalene	5	0.39	ug/kg
8270C-SIM PAH	3541	Soil	Acenaphthene	5	0.23	ug/kg
8270C-SIM PAH	3541	Soil	Acenaphthylene	5	0.24	ug/kg
8270C-SIM PAH	3541	Soil	Anthracene	5	0.47	ug/kg
8270C-SIM PAH	3541	Soil	Benz(a)anthracene	5	0.48	ug/kg
8270C-SIM PAH	3541	Soil	Benzo(a)pyrene	5	0.14	ug/kg
8270C-SIM PAH	3541	Soil	Benzo(b)fluoranthene	5	0.25	ug/kg
8270C-SIM PAH	3541	Soil	Benzo(e)pyrene	5	0.18	ug/kg
8270C-SIM PAH	3541	Soil	Benzo(g,h,i)perylene	5	0.64	ug/kg
8270C-SIM PAH	3541	Soil	Benzo(k)fluoranthene	5	0.15	ug/kg
8270C-SIM PAH	3541	Soil	Biphenyl	5	0.40	ug/kg
8270C-SIM PAH	3541	Soil	Carbazole	5	2.3	ug/kg
8270C-SIM PAH	3541	Soil	Chrysene	5	0.25	ug/kg
8270C-SIM PAH	3541	Soil	Dibenz(a,h)anthracene	5	0.28	ug/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C-SIM PAH	3541	Soil	Dibenzofuran	5	0.59	ug/kg
8270C-SIM PAH	3541	Soil	Dibenzothiophene	5	0.21	ug/kg
8270C-SIM PAH	3541	Soil	Fluoranthene	5	0.61	ug/kg
8270C-SIM PAH	3541	Soil	Fluorene	5	0.50	ug/kg
8270C-SIM PAH	3541	Soil	Indeno(1,2,3-cd)pyrene	5	0.16	ug/kg
8270C-SIM PAH	3541	Soil	Naphthalene	5	0.37	ug/kg
8270C-SIM PAH	3541	Soil	Pentachlorophenol	200	2.9	ug/kg
8270C-SIM PAH	3541	Soil	Perylene	5	0.32	ug/kg
8270C-SIM PAH	3541	Soil	Phenanthrene	5	0.75	ug/kg
8270C-SIM PAH	3541	Soil	Pyrene	5	0.37	ug/kg
8270C-SIM PAH	3520C	Water	1-Methylnaphthalene	0.02	0.0035	ug/L
8270C-SIM PAH	3520C	Water	1-Methylphenanthrene	0.02	0.0041	ug/L
8270C-SIM PAH	3520C	Water	2,3,5-Trimethylnaphthalene	0.02	0.0050	ug/L
8270C-SIM PAH	3520C	Water	2,6-Dimethylnaphthalene	0.02	0.0022	ug/L
8270C-SIM PAH	3520C	Water	2-Methylnaphthalene	0.02	0.0023	ug/L
8270C-SIM PAH	3520C	Water	Acenaphthene	0.02	0.0044	ug/L
8270C-SIM PAH	3520C	Water	Acenaphthylene	0.02	0.0034	ug/L
8270C-SIM PAH	3520C	Water	Anthracene	0.02	0.0036	ug/L
8270C-SIM PAH	3520C	Water	Benz(a)anthracene	0.02	0.0026	ug/L
8270C-SIM PAH	3520C	Water	Benzo(a)pyrene	0.02	0.0043	ug/L
8270C-SIM PAH	3520C	Water	Benzo(b)fluoranthene	0.02	0.0023	ug/L
8270C-SIM PAH	3520C	Water	Benzo(e)pyrene	0.02	0.0040	ug/L
8270C-SIM PAH	3520C	Water	Benzo(g,h,i)perylene	0.02	0.0029	ug/L
8270C-SIM PAH	3520C	Water	Benzo(k)fluoranthene	0.02	0.0025	ug/L
8270C-SIM PAH	3520C	Water	Biphenyl	0.02	0.0024	ug/L
8270C-SIM PAH	3520C	Water	Carbazole	0.02	0.0045	ug/L
8270C-SIM PAH	3520C	Water	Chrysene	0.02	0.0034	ug/L
8270C-SIM PAH	3520C	Water	Dibenz(a,h)anthracene	0.02	0.0025	ug/L
8270C-SIM PAH	3520C	Water	Dibenzofuran	0.02	0.0046	ug/L
8270C-SIM PAH	3520C	Water	Dibenzothiophene	0.02	0.0038	ug/L
8270C-SIM PAH	3520C	Water	Fluoranthene	0.02	0.0044	ug/L
8270C-SIM PAH	3520C	Water	Fluorene	0.02	0.0038	ug/L
8270C-SIM PAH	3520C	Water	Indeno(1,2,3-cd)pyrene	0.02	0.0026	ug/L
8270C-SIM PAH	3520C	Water	Naphthalene	0.02	0.0030	ug/L
8270C-SIM PAH	3520C	Water	Pentachlorophenol	1	0.017	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C-SIM PAH	3520C	Water	Perylene	0.02	0.0050	ug/L
8270C-SIM PAH	3520C	Water	Phenanthrene	0.02	0.0050	ug/L
8270C-SIM PAH	3520C	Water	Pyrene	0.02	0.0035	ug/L
8310	3550B	Soil	2-Methylnaphthalene/Dibenzofuran	0.1	0.032	mg/kg
8310	3550B	Soil	Acenaphthene	0.1	0.023	mg/kg
8310	3550B	Soil	Acenaphthylene	0.1	0.029	mg/kg
8310	3550B	Soil	Anthracene	0.01	0.00069	mg/kg
8310	3550B	Soil	Benz(a)anthracene	0.01	0.0013	mg/kg
8310	3550B	Soil	Benzo(a)pyrene	0.01	0.0010	mg/kg
8310	3550B	Soil	Benzo(b)fluoranthene	0.02	0.00071	mg/kg
8310	3550B	Soil	Benzo(g,h,i)perylene	0.02	0.0025	mg/kg
8310	3550B	Soil	Benzo(k)fluoranthene	0.01	0.00096	mg/kg
8310	3550B	Soil	Chrysene	0.01	0.00092	mg/kg
8310	3550B	Soil	Dibenz(a,h)anthracene	0.02	0.0021	mg/kg
8310	3550B	Soil	Fluoranthene	0.02	0.0014	mg/kg
8310	3550B	Soil	Fluorene	0.02	0.0025	mg/kg
8310	3550B	Soil	Indeno(1,2,3-cd)pyrene	0.01	0.0023	mg/kg
8310	3550B	Soil	Naphthalene	0.1	0.021	mg/kg
8310	3550B	Soil	Phenanthrene	0.01	0.0023	mg/kg
8310	3550B	Soil	Pyrene	0.02	0.00084	mg/kg
8310	3510C	Water	2-Methylnaphthalene/Dibenzofuran	1.0	0.14	ug/L
8310	3510C	Water	Acenaphthene	1.0	0.15	ug/L
8310	3510C	Water	Acenaphthylene	1.0	0.12	ug/L
8310	3510C	Water	Anthracene	0.1	0.0099	ug/L
8310	3510C	Water	Benz(a)anthracene	0.1	0.021	ug/L
8310	3510C	Water	Benzo(a)pyrene	0.1	0.012	ug/L
8310	3510C	Water	Benzo(b)fluoranthene	0.2	0.067	ug/L
8310	3510C	Water	Benzo(g,h,i)perylene	0.2	0.036	ug/L
8310	3510C	Water	Benzo(k)fluoranthene	0.1	0.033	ug/L
8310	3510C	Water	Chrysene	0.1	0.021	ug/L
8310	3510C	Water	Dibenz(a,h)anthracene	0.2	0.035	ug/L
8310	3510C	Water	Fluoranthene	0.2	0.037	ug/L
8310	3510C	Water	Fluorene	0.2	0.018	ug/L
8310	3510C	Water	Indeno(1,2,3-cd)pyrene	0.1	0.034	ug/L
8310	3510C	Water	Naphthalene	1.0	0.13	ug/L

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8310	3510C	Water	Phenanthrene	0.1	0.013	ug/L
8310	3510C	Water	Pyrene	0.2	0.024	ug/L
8315	Method	Soil	Acetaldehyde	2	1	mg/kg
8315	Method	Soil	Formaldehyde	2	0.6	mg/kg
8315	Method	Water	Acetaldehyde	100	12	ug/L
8315	Method	Water	Formaldehyde	100	25	ug/L
8330	Method	Soil	1,3,5-Trinitrobenzene	2.0	0.1	mg/kg
8330	Method	Soil	1,3-Dinitrobenzene	2.0	0.065	mg/kg
8330	Method	Soil	2,4,6-Trinitrotoluene	2.0	0.066	mg/kg
8330	Method	Soil	2,4-Dinitrotoluene	2.0	0.09	mg/kg
8330	Method	Soil	2,6-Dinitrotoluene	2.0	0.085	mg/kg
8330	Method	Soil	2-Amino-4,6-dinitrotoluene	2.0	0.072	mg/kg
8330	Method	Soil	2-Nitrotoluene	2.0	0.15	mg/kg
8330	Method	Soil	3-Nitrotoluene	2.0	0.13	mg/kg
8330	Method	Soil	4-Amino-2,6-dinitrotoluene	2.0	0.068	mg/kg
8330	Method	Soil	4-Nitrotoluene	2.0	0.12	mg/kg
8330	Method	Soil	HMX	2.0	0.12	mg/kg
8330	Method	Soil	Nitrobenzene	2.0	0.058	mg/kg
8330	Method	Soil	RDX	2.0	0.18	mg/kg
8330	Method	Soil	TETRYL	2.0	0.11	mg/kg
8330	Method	Water	1,3,5-Trinitrobenzene	2.0	0.16	ug/L
8330	Method	Water	1,3-Dinitrobenzene	2.0	0.11	ug/L
8330	Method	Water	2,4,6-Trinitrotoluene	2.0	0.18	ug/L
8330	Method	Water	2,4-Dinitrotoluene	2.0	0.16	ug/L
8330	Method	Water	2,6-Dinitrotoluene	2.0	0.18	ug/L
8330	Method	Water	2-Amino-4,6-dinitrotoluene	2.0	0.29	ug/L
8330	Method	Water	2-Nitrotoluene	2.0	0.28	ug/L
8330	Method	Water	3-Nitrotoluene	2.0	0.21	ug/L
8330	Method	Water	4-Amino-2,6-dinitrotoluene	2.0	0.14	ug/L
8330	Method	Water	4-Nitrotoluene	2.0	0.29	ug/L
8330	Method	Water	HMX	2.0	0.22	ug/L
8330	Method	Water	Nitrobenzene	2.0	0.20	ug/L
8330	Method	Water	RDX	2.0	0.25	ug/L
8330	Method	Water	TETRYL	2.0	0.16	ug/L
8332	Method	Soil	Nitroglycerin	2	0.28	mg/kg

SEMIVOLATILE ORGANIC COMPOUNDS (SOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8332	Method	Soil	PETN	2	0.42	mg/kg
8332	Method	Water	Nitroglycerin	2	0.36	ug/L
8332	Method	Water	PETN	2	0.16	ug/L
AK 102	Method	Soil	Diesel Range Petroleum Hydrocarbons	20	2.8	mg/kg
8015	3550B	Soil	Diesel Range Petroleum Hydrocarbons	10	2.3	mg/kg
NWTPH-Dx	Method	Soil	Diesel Range Petroleum Hydrocarbons	25	2.6	mg/kg
AK 102	Method	Water	Diesel Range Petroleum Hydrocarbons	800	52	ug/L
8015	3510C	Water	Diesel Range Petroleum Hydrocarbons	100	49	ug/L
NWTPH-Dx	Method	Water	Diesel Range Petroleum Hydrocarbons	250	47	ug/L
AK 103	3550B	Soil	Residual Range Petroleum Hydrocarbons	100	15	mg/kg
8015	3510C	Water	Residual Range Petroleum Hydrocarbons	100	76	ug/L
NWTPH-Dx	Method	Water	Residual Range Petroleum Hydrocarbons	500	19	ug/L
NCASI 94.03	Method	Water	Methanol	0.5	0.23	ug/mL
NCASI 99.01	Method	Water	Acetaldehyde	1	0.15	ug/mL
NCASI 99.01	Method	Water	Methanol	0.5	0.17	ug/mL
NCASI 99.01	Method	Water	Methyl Ethyl Ketone	1	0.17	ug/mL
NCASI 99.01	Method	Water	Propionitrile	1	0.16	ug/mL
NCASI 99.02	Method	Impinger	Acrolein	50	5	ug
NCASI 99.02	Method	Impinger	Acetaldehyde	50	17	ug
NCASI 99.02	Method	Impinger	Methanol	50	16	ug
NCASI 99.02	Method	Impinger	Methyl Ethyl Ketone	50	7.2	ug
NCASI 99.02	Method	Impinger	Propionaldehyde	50	5.8	ug

a Method Detection Limits are subject to change as new MDL studies are completed.

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
624	Method	Water	1,1,1-Trichloroethane (TCA)	5	0.12	ug/L
624	Method	Water	1,1,2,2-Tetrachloroethane	5	0.11	ug/L
624	Method	Water	1,1,2-Trichloroethane	5	0.16	ug/L
624	Method	Water	1,1-Dichloroethane	5	0.11	ug/L
624	Method	Water	1,1-Dichloroethene	5	0.15	ug/L
624	Method	Water	1,2-Dichlorobenzene	5	0.13	ug/L
624	Method	Water	1,2-Dichloroethane (EDC)	5	0.12	ug/L
624	Method	Water	1,2-Dichloropropane	5	0.17	ug/L
624	Method	Water	1,3-Dichlorobenzene	5	0.16	ug/L
624	Method	Water	1,4-Dichlorobenzene	5	0.15	ug/L
624	Method	Water	2-Butanone (MEK)	20	2.6	ug/L
624	Method	Water	2-Chloroethyl Vinyl Ether	10	0.29	ug/L
624	Method	Water	2-Hexanone	20	1.9	ug/L
624	Method	Water	Acetone	20	2.1	ug/L
624	Method	Water	Acrolein	50	3.3	ug/L
624	Method	Water	Acrylonitrile	10	0.61	ug/L
624	Method	Water	Benzene	5	0.14	ug/L
624	Method	Water	Bromodichloromethane	5	0.12	ug/L
624	Method	Water	Bromoform	5	0.37	ug/L
624	Method	Water	Bromomethane	5	0.2	ug/L
624	Method	Water	Carbon Disulfide	5	0.13	ug/L
624	Method	Water	Carbon Tetrachloride	5	0.047	ug/L
624	Method	Water	Chlorobenzene	5	0.098	ug/L
624	Method	Water	Chloroethane	5	0.15	ug/L
624	Method	Water	Chloroform	5	0.11	ug/L
624	Method	Water	Chloromethane	5	0.23	ug/L
624	Method	Water	cis-1,2-Dichloroethene	5	0.15	ug/L
624	Method	Water	cis-1,3-Dichloropropene	5	0.13	ug/L
624	Method	Water	Dibromochloromethane	5	0.13	ug/L
624	Method	Water	Dichlorodifluoromethane	5	0.19	ug/L
624	Method	Water	Ethylbenzene	5	0.11	ug/L
624	Method	Water	m,p-Xylenes	5	0.26	ug/L

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
624	Method	Water	Methylene Chloride	5	0.1	ug/L
624	Method	Water	o-Xylene	5	0.13	ug/L
624	Method	Water	Styrene	5	0.14	ug/L
624	Method	Water	Tetrachloroethene (PCE)	5	0.098	ug/L
624	Method	Water	Toluene	5	0.18	ug/L
624	Method	Water	trans-1,2-Dichloroethene	5	0.15	ug/L
624	Method	Water	trans-1,3-Dichloropropene	5	0.1	ug/L
624	Method	Water	Trichloroethene (TCE)	5	0.11	ug/L
624	Method	Water	Trichlorofluoromethane	5	0.11	ug/L
624	Method	Water	Trichlorotrifluoroethane	5	0.11	ug/L
624	Method	Water	Vinyl Acetate	10	0.57	ug/L
624	Method	Water	Vinyl Chloride	5	0.16	ug/L
8021B	5035/5030B	Soil	Benzene	0.05	0.016	mg/kg
8021B	5035/5030B	Soil	Ethylbenzene	0.10	0.016	mg/kg
8021B	5035/5030B	Soil	m,p-Xylenes	0.10	0.042	mg/kg
8021B	5035/5030B	Soil	o-Xylene	0.10	0.014	mg/kg
8021B	5035/5030B	Soil	Toluene	0.10	0.01	mg/kg
8021B	5030B	Water	Benzene	0.5	0.099	ug/L
8021B	5030B	Water	Ethylbenzene	1.0	0.10	ug/L
8021B	5030B	Water	m,p-Xylenes	1.0	0.21	ug/L
8021B	5030B	Water	o-Xylene	1.0	0.10	ug/L
8021B	5030B	Water	Toluene	1.0	0.098	ug/L
8260B	5030A/5035	Soil-low	1,1,1,2-Tetrachloroethane	5.0	0.076	ug/kg
8260B	5030A/5035	Soil-low	1,1,1-Trichloroethane (TCA)	5.0	0.066	ug/kg
8260B	5030A/5035	Soil-low	1,1,2,2-Tetrachloroethane	5.0	0.11	ug/kg
8260B	5030A/5035	Soil-low	1,1,2-Trichloroethane	5.0	0.11	ug/kg
8260B	5030A/5035	Soil-low	1,1-Dichloroethane	5.0	0.068	ug/kg
8260B	5030A/5035	Soil-low	1,1-Dichloroethene	5.0	0.057	ug/kg
8260B	5030A/5035	Soil-low	1,1-Dichloropropene	5.0	0.062	ug/kg
8260B	5030A/5035	Soil-low	1,2,3-Trichlorobenzene	20	0.18	ug/kg
8260B	5030A/5035	Soil-low	1,2,3-Trichloropropane	5.0	0.29	ug/kg

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8260B	5030A/5035	Soil-low	1,2,4-Trichlorobenzene	20	0.19	ug/kg
8260B	5030A/5035	Soil-low	1,2,4-Trimethylbenzene	20	0.07	ug/kg
8260B	5030A/5035	Soil-low	1,2-Dibromo-3-chloropropane	20	0.34	ug/kg
8260B	5030A/5035	Soil-low	1,2-Dibromoethane (EDB)	20	0.093	ug/kg
8260B	5030A/5035	Soil-low	1,2-Dichlorobenzene	5.0	0.065	ug/kg
8260B	5030A/5035	Soil-low	1,2-Dichloroethane (EDC)	5.0	0.057	ug/kg
8260B	5030A/5035	Soil-low	1,2-Dichloropropane	5.0	0.094	ug/kg
8260B	5030A/5035	Soil-low	1,3,5-Trimethylbenzene	20	0.069	ug/kg
8260B	5030A/5035	Soil-low	1,3-Dichlorobenzene	5.0	0.056	ug/kg
8260B	5030A/5035	Soil-low	1,3-Dichloropropane	5.0	0.083	ug/kg
8260B	5030A/5035	Soil-low	1,4-Dichlorobenzene	5.0	0.1	ug/kg
8260B	5030A/5035	Soil-low	1,4-Dioxane	250	13	ug/kg
8260B	5030A/5035	Soil-low	1-Chlorohexane	5.0	0.1	ug/kg
8260B	5030A/5035	Soil-low	2,2-Dichloropropane	5.0	0.088	ug/kg
8260B	5030A/5035	Soil-low	2-Butanone (MEK)	20	1.1	ug/kg
8260B	5030A/5035	Soil-low	2-Chloroethyl Vinyl Ether	10	0.31	ug/kg
8260B	5030A/5035	Soil-low	2-Chlorotoluene	20	0.051	ug/kg
8260B	5030A/5035	Soil-low	2-Hexanone	20	0.59	ug/kg
8260B	5030A/5035	Soil-low	2-Nitropropane	20	0.093	ug/kg
8260B	5030A/5035	Soil-low	4-Chlorotoluene	20	0.082	ug/kg
8260B	5030A/5035	Soil-low	4-Isopropyltoluene	20	0.066	ug/kg
8260B	5030A/5035	Soil-low	4-Methyl-2-pentanone (MIBK)	20	0.22	ug/kg
8260B	5030A/5035	Soil-low	Acetone	20	1.8	ug/kg
8260B	5030A/5035	Soil-low	Acetonitrile	100	13	ug/kg
8260B	5030A/5035	Soil-low	Acrolein	100	1.7	ug/kg
8260B	5030A/5035	Soil-low	Acrylonitrile	20	0.46	ug/kg
8260B	5030A/5035	Soil-low	Benzene	5.0	0.079	ug/kg
8260B	5030A/5035	Soil-low	Bromobenzene	5.0	0.14	ug/kg
8260B	5030A/5035	Soil-low	Bromochloromethane	5.0	0.18	ug/kg
8260B	5030A/5035	Soil-low	Bromodichloromethane	5.0	0.11	ug/kg
8260B	5030A/5035	Soil-low	Bromoform	5.0	0.19	ug/kg
8260B	5030A/5035	Soil-low	Bromomethane	5.0	0.51	ug/kg

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8260B	5030A/5035	Soil-low	Carbon Disulfide	5.0	0.057	ug/kg
8260B	5030A/5035	Soil-low	Carbon Tetrachloride	5.0	0.072	ug/kg
8260B	5030A/5035	Soil-low	Chlorobenzene	5.0	0.051	ug/kg
8260B	5030A/5035	Soil-low	Chloroethane	5.0	0.24	ug/kg
8260B	5030A/5035	Soil-low	Chloroform	5.0	0.063	ug/kg
8260B	5030A/5035	Soil-low	Chloromethane	5.0	0.11	ug/kg
8260B	5030A/5035	Soil-low	Chloroprene	20	0.1	ug/kg
8260B	5030A/5035	Soil-low	cis-1,2-Dichloroethene	5.0	0.1	ug/kg
8260B	5030A/5035	Soil-low	cis-1,3-Dichloropropene	5.0	0.056	ug/kg
8260B	5030A/5035	Soil-low	cis-1,4-Dichloro-2-butene	20	0.3	ug/kg
8260B	5030A/5035	Soil-low	Cyclohexane	5.0	0.32	ug/kg
8260B	5030A/5035	Soil-low	Dibromochloromethane	5.0	0.096	ug/kg
8260B	5030A/5035	Soil-low	Dibromomethane	5.0	0.15	ug/kg
8260B	5030A/5035	Soil-low	Dichlorodifluoromethane	5.0	0.066	ug/kg
8260B	5030A/5035	Soil-low	Diisopropyl Ether	10	0.07	ug/kg
8260B	5030A/5035	Soil-low	Ethyl Acetate	20	1.6	ug/kg
8260B	5030A/5035	Soil-low	Ethyl Methacrylate	20	0.19	ug/kg
8260B	5030A/5035	Soil-low	Ethylbenzene	5.0	0.065	ug/kg
8260B	5030A/5035	Soil-low	Ethylene Oxide	100	12	ug/kg
8260B	5030A/5035	Soil-low	Hexachlorobutadiene	20	0.11	ug/kg
8260B	5030A/5035	Soil-low	Iodomethane (Methyl Iodide)	20	0.35	ug/kg
8260B	5030A/5035	Soil-low	Isobutanol	200	2.6	ug/kg
8260B	5030A/5035	Soil-low	Isopropylbenzene	20	0.05	ug/kg
8260B	5030A/5035	Soil-low	m,p-Xylenes	5.0	0.15	ug/kg
8260B	5030A/5035	Soil-low	Methacrylonitrile	20	0.33	ug/kg
8260B	5030A/5035	Soil-low	Methyl Acetate	5.0	0.59	ug/kg
8260B	5030A/5035	Soil-low	Methyl Methacrylate	20	0.19	ug/kg
8260B	5030A/5035	Soil-low	Methyl tert-Butyl Ether	5.0	0.064	ug/kg
8260B	5030A/5035	Soil-low	Methylcyclohexane	5.0	0.21	ug/kg
8260B	5030A/5035	Soil-low	Methylene Chloride	10	0.14	ug/kg
8260B	5030A/5035	Soil-low	Naphthalene	20	0.14	ug/kg
8260B	5030A/5035	Soil-low	n-Butylbenzene	20	0.096	ug/kg

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8260B	5030A/5035	Soil-low	n-Hexane	10	0.16	ug/kg
8260B	5030A/5035	Soil-low	n-Octane	10	4	ug/kg
8260B	5030A/5035	Soil-low	n-Propylbenzene	20	0.054	ug/kg
8260B	5030A/5035	Soil-low	o-Xylene	5.0	0.057	ug/kg
8260B	5030A/5035	Soil-low	Propionitrile	20	0.79	ug/kg
8260B	5030A/5035	Soil-low	Propylene Oxide	50	10	ug/kg
8260B	5030A/5035	Soil-low	sec-Butylbenzene	20	0.072	ug/kg
8260B	5030A/5035	Soil-low	Styrene	5.0	0.067	ug/kg
8260B	5030A/5035	Soil-low	tert-Amyl Methyl Ether	10	0.2	ug/kg
8260B	5030A/5035	Soil-low	tert-Butyl Alcohol	50	1.7	ug/kg
8260B	5030A/5035	Soil-low	tert-Butyl Ethyl Ether	10	0.09	ug/kg
8260B	5030A/5035	Soil-low	tert-Butylbenzene	20	0.07	ug/kg
8260B	5030A/5035	Soil-low	Tetrachloroethene (PCE)	5.0	0.076	ug/kg
8260B	5030A/5035	Soil-low	Toluene	5.0	0.13	ug/kg
8260B	5030A/5035	Soil-low	trans-1,2-Dichloroethene	5.0	0.084	ug/kg
8260B	5030A/5035	Soil-low	trans-1,3-Dichloropropene	5.0	0.093	ug/kg
8260B	5030A/5035	Soil-low	trans-1,4-Dichloro-2-butene	20	0.41	ug/kg
8260B	5030A/5035	Soil-low	Trichloroethene (TCE)	5.0	0.07	ug/kg
8260B	5030A/5035	Soil-low	Trichlorofluoromethane	5.0	0.11	ug/kg
8260B	5030A/5035	Soil-low	Trichlorotrifluoroethane	5.0	0.14	ug/kg
8260B	5030A/5035	Soil-low	Vinyl Acetate	20	0.95	ug/kg
8260B	5030A/5035	Soil-low	Vinyl Chloride	5.0	0.094	ug/kg
8260B	5035/5030B	Soil-high	1,1,1,2-Tetrachloroethane	.05	0.0111	mg/kg
8260B	5035/5030B	Soil-high	1,1,1-Trichloroethane (TCA)	.05	0.0111	mg/kg
8260B	5035/5030B	Soil-high	1,1,2,2-Tetrachloroethane	.05	0.0138	mg/kg
8260B	5035/5030B	Soil-high	1,1,2-Trichloroethane	.05	0.0104	mg/kg
8260B	5035/5030B	Soil-high	1,1-Dichloroethane	.05	0.00906	mg/kg
8260B	5035/5030B	Soil-high	1,1-Dichloroethene	.05	0.0198	mg/kg
8260B	5035/5030B	Soil-high	1,1-Dichloropropene	.05	0.0128	mg/kg
8260B	5035/5030B	Soil-high	1,2,3-Trichlorobenzene	0.2	0.0326	mg/kg
8260B	5035/5030B	Soil-high	1,2,3-Trichloropropane	.05	0.0213	mg/kg
8260B	5035/5030B	Soil-high	1,2,4-Trichlorobenzene	0.2	0.0218	mg/kg

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8260B	5035/5030B	Soil-high	1,2,4-Trimethylbenzene	0.2	0.0141	mg/kg
8260B	5035/5030B	Soil-high	1,2-Dibromo-3-chloropropane	0.2	0.0991	mg/kg
8260B	5035/5030B	Soil-high	1,2-Dibromoethane (EDB)	0.2	0.00073	mg/kg
8260B	5035/5030B	Soil-high	1,2-Dichlorobenzene	.05	0.00847	mg/kg
8260B	5035/5030B	Soil-high	1,2-Dichloroethane (EDC)	.05	0.0114	mg/kg
8260B	5035/5030B	Soil-high	1,2-Dichloropropane	.05	0.019	mg/kg
8260B	5035/5030B	Soil-high	1,3,5-Trichlorobenzene	0.2	0.0343	mg/kg
8260B	5035/5030B	Soil-high	1,3,5-Trimethylbenzene	0.2	.0121	mg/kg
8260B	5035/5030B	Soil-high	1,3-Dichlorobenzene	.05	0.0102	mg/kg
8260B	5035/5030B	Soil-high	1,3-Dichloropropane	.05	0.0087	mg/kg
8260B	5035/5030B	Soil-high	1,4-Dichlorobenzene	.05	.00869	mg/kg
8260B	5035/5030B	Soil-high	1,4-Dioxane	25	10	mg/kg
8260B	5035/5030B	Soil-high	1-Chlorohexane	.05	0.0118	mg/kg
8260B	5035/5030B	Soil-high	2,2-Dichloropropane	.05	0.0174	mg/kg
8260B	5035/5030B	Soil-high	2-Butanone (MEK)	2.0	0.322	mg/kg
8260B	5035/5030B	Soil-high	2-Chloroethyl Vinyl Ether	0.5	0.043	mg/kg
8260B	5035/5030B	Soil-high	2-Chlorotoluene	0.2	0.0111	mg/kg
8260B	5035/5030B	Soil-high	2-Hexanone	2.0	0.396	mg/kg
8260B	5035/5030B	Soil-high	2-Nitropropane	0.5	0.32	mg/kg
8260B	5035/5030B	Soil-high	3-Chloro-1-propene	0.5	0.0127	mg/kg
8260B	5035/5030B	Soil-high	4-Chlorotoluene	0.2	0.00884	mg/kg
8260B	5035/5030B	Soil-high	4-Isopropyltoluene	0.2	0.0128	mg/kg
8260B	5035/5030B	Soil-high	4-Methyl-2-pentanone (MIBK)	2.0	0.34	mg/kg
8260B	5035/5030B	Soil-high	Acetone	2.0	0.24	mg/kg
8260B	5035/5030B	Soil-high	Acetonitrile	0.5	0.62	mg/kg
8260B	5035/5030B	Soil-high	Acrolein	2.0	0.17	mg/kg
8260B	5035/5030B	Soil-high	Acrylonitrile	2.0	0.032	mg/kg
8260B	5035/5030B	Soil-high	Benzene	.05	0.0105	mg/kg
8260B	5035/5030B	Soil-high	Bromobenzene	0.2	0.01	mg/kg
8260B	5035/5030B	Soil-high	Bromochloromethane	.05	0.0126	mg/kg
8260B	5035/5030B	Soil-high	Bromodichloromethane	.05	0.0087	mg/kg
8260B	5035/5030B	Soil-high	Bromoform	.05	0.0279	mg/kg

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8260B	5035/5030B	Soil-high	Bromomethane	.05	0.0217	mg/kg
8260B	5035/5030B	Soil-high	Carbon Disulfide	.05	0.0159	mg/kg
8260B	5035/5030B	Soil-high	Carbon Tetrachloride	.05	0.013	mg/kg
8260B	5035/5030B	Soil-high	Chlorobenzene	.05	0.00933	mg/kg
8260B	5035/5030B	Soil-high	Chloroethane	.05	0.0173	mg/kg
8260B	5035/5030B	Soil-high	Chloroform	.05	0.00958	mg/kg
8260B	5035/5030B	Soil-high	Chloromethane	.05	0.0136	mg/kg
8260B	5035/5030B	Soil-high	Chloroprene	1.0	0.056	mg/kg
8260B	5035/5030B	Soil-high	cis-1,2-Dichloroethene	.05	0.0116	mg/kg
8260B	5035/5030B	Soil-high	cis-1,3-Dichloropropene	.05	0.0089	mg/kg
8260B	5035/5030B	Soil-high	cis-1,4-Dichloro-2-butene	1.0	0.14	mg/kg
8260B	5035/5030B	Soil-high	Cyclohexane	0.1	0.010	mg/kg
8260B	5035/5030B	Soil-high	Dibromochloromethane	.05	0.00817	mg/kg
8260B	5035/5030B	Soil-high	Dibromomethane	.05	0.017	mg/kg
8260B	5035/5030B	Soil-high	Dichlorodifluoromethane	.05	0.0166	mg/kg
8260B	5035/5030B	Soil-high	Dichlorofluoromethane (CFC 21)	.05	0.016	mg/kg
8260B	5035/5030B	Soil-high	Ethyl Acetate	0.5	0.1	mg/kg
8260B	5035/5030B	Soil-high	Ethyl Ether	0.1	0.0055	mg/kg
8260B	5035/5030B	Soil-high	Ethyl Methacrylate	0.2	0.01	mg/kg
8260B	5035/5030B	Soil-high	Ethylbenzene	.05	0.00974	mg/kg
8260B	5035/5030B	Soil-high	Hexachlorobutadiene	0.2	0.0380	mg/kg
8260B	5035/5030B	Soil-high	Iodomethane	0.5	0.065	mg/kg
8260B	5035/5030B	Soil-high	Isobutyl Alcohol	50	0.9	mg/kg
8260B	5035/5030B	Soil-high	Isopropylbenzene	0.2	0.0093	mg/kg
8260B	5035/5030B	Soil-high	m,p-Xylenes	.05	0.0186	mg/kg
8260B	5035/5030B	Soil-high	Methacrylonitrile	0.5	0.035	mg/kg
8260B	5035/5030B	Soil-high	Methyl Acetate	0.1	0.0094	mg/kg
8260B	5035/5030B	Soil-high	Methyl Methacrylate	0.5	0.016	mg/kg
8260B	5035/5030B	Soil-high	Methyl tert-Butyl Ether	.05	0.015	mg/kg
8260B	5035/5030B	Soil-high	Methylcyclohexane	0.1	0.0059	mg/kg
8260B	5035/5030B	Soil-high	Methylene Chloride	0.2	0.0193	mg/kg
8260B	5035/5030B	Soil-high	Naphthalene	0.2	0.0285	mg/kg

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8260B	5035/5030B	Soil-high	n-Butylbenzene	0.2	0.0221	mg/kg
8260B	5035/5030B	Soil-high	n-Heptane	0.1	0.1	mg/kg
8260B	5035/5030B	Soil-high	n-Hexane	0.1	0.0180	mg/kg
8260B	5035/5030B	Soil-high	n-Octane	0.1	0.029	mg/kg
8260B	5035/5030B	Soil-high	n-Propylbenzene	0.2	0.00968	mg/kg
8260B	5035/5030B	Soil-high	o-Xylene	.05	0.00785	mg/kg
8260B	5035/5030B	Soil-high	Propionitrile	0.5	0.11	mg/kg
8260B	5035/5030B	Soil-high	sec-Butylbenzene	0.2	0.0127	mg/kg
8260B	5035/5030B	Soil-high	Styrene	.05	0.00943	mg/kg
8260B	5035/5030B	Soil-high	tert-Butyl Alcohol	2.0	0.28	mg/kg
8260B	5035/5030B	Soil-high	tert-Butyl Ethyl Ether	0.05	0.0099	mg/kg
8260B	5035/5030B	Soil-high	tert-Butylbenzene	0.2	0.0122	mg/kg
8260B	5035/5030B	Soil-high	Tetrachloroethene (PCE)	.05	0.014	mg/kg
8260B	5035/5030B	Soil-high	Tetrahydrofuran	1.0	0.5	mg/kg
8260B	5035/5030B	Soil-high	Toluene	.05	0.00975	mg/kg
8260B	5035/5030B	Soil-high	trans-1,2-Dichloroethene	.05	0.0139	mg/kg
8260B	5035/5030B	Soil-high	trans-1,3-Dichloropropene	.05	0.0091	mg/kg
8260B	5035/5030B	Soil-high	trans-1,4-Dichloro-2-butene	1.0	0.0597	mg/kg
8260B	5035/5030B	Soil-high	Trichloroethene (TCE)	.05	0.0188	mg/kg
8260B	5035/5030B	Soil-high	Trichlorofluoromethane	.05	0.017	mg/kg
8260B	5035/5030B	Soil-high	Trichlorotrifluoroethane	.05	0.02	mg/kg
8260B	5035/5030B	Soil-high	Vinyl Acetate	0.5	0.0387	mg/kg
8260B	5035/5030B	Soil-high	Vinyl Chloride	.05	0.0211	mg/kg
8260B	5030B	Water	1,1,1,2-Tetrachloroethane	0.5	0.047	ug/L
8260B	5030B	Water	1,1,1-Trichloroethane (TCA)	0.5	0.050	ug/L
8260B	5030B	Water	1,1,2,2-Tetrachloroethane	0.5	0.064	ug/L
8260B	5030B	Water	1,1,2-Trichloroethane	0.5	0.061	ug/L
8260B	5030B	Water	1,1-Dichloroethane	0.5	0.042	ug/L
8260B	5030B	Water	1,1-Dichloroethene	0.5	0.10	ug/L
8260B	5030B	Water	1,1-Dichloropropene	0.5	0.051	ug/L
8260B	5030B	Water	1,2,3-Trichlorobenzene	2	0.10	ug/L
8260B	5030B	Water	1,2,3-Trichloropropane	0.5	0.14	ug/L

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8260B	5030B	Water	1,2,4-Trichlorobenzene	2	0.13	ug/L
8260B	5030B	Water	1,2,4-Trimethylbenzene	2	0.037	ug/L
8260B	5030B	Water	1,2-Dibromo-3-chloropropane	2	0.22	ug/L
8260B	5030B	Water	1,2-Dibromoethane (EDB)	2	0.084	ug/L
8260B	5030B	Water	1,2-Dichlorobenzene	0.5	0.044	ug/L
8260B	5030B	Water	1,2-Dichloroethane (EDC)	0.5	0.073	ug/L
8260B	5030B	Water	1,2-Dichloropropane	0.5	0.042	ug/L
8260B	5030B	Water	1,3,5-Trichlorobenzene	5	0.10	ug/L
8260B	5030B	Water	1,3,5-Trimethylbenzene	2	0.042	ug/L
8260B	5030B	Water	1,3-Dichlorobenzene	0.5	0.041	ug/L
8260B	5030B	Water	1,3-Dichloropropane	0.5	0.032	ug/L
8260B	5030B	Water	1,4-Dichlorobenzene	0.5	0.054	ug/L
8260B	5030B	Water	1,4-Dioxane	100	13	ug/L
8260B	5030B	Water	1-Chlorohexane	0.5	0.057	ug/L
8260B	5030B	Water	2,2-Dichloropropane	0.5	0.050	ug/L
8260B	5030B	Water	2-Butanone (MEK)	20	3.8	ug/L
8260B	5030B	Water	2-Chloroethyl Vinyl Ether	5	0.19	ug/L
8260B	5030B	Water	2-Chlorotoluene	2	0.035	ug/L
8260B	5030B	Water	2-Hexanone	20	2.9	ug/L
8260B	5030B	Water	2-Nitropropane	5	0.91	ug/L
8260B	5030B	Water	3-Chloro-1-propene	5	0.19	ug/L
8260B	5030B	Water	4-Chlorotoluene	2	0.025	ug/L
8260B	5030B	Water	4-Isopropyltoluene	2	0.044	ug/L
8260B	5030B	Water	4-Methyl-2-pentanone (MIBK)	20	3.0	ug/L
8260B	5030B	Water	Acetone	20	2.5	ug/L
8260B	5030B	Water	Acetonitrile	50	7.3	ug/L
8260B	5030B	Water	Acrolein	20	2.0	ug/L
8260B	5030B	Water	Acrylonitrile	5	0.31	ug/L
8260B	5030B	Water	Benzene	0.5	0.045	ug/L
8260B	5030B	Water	Bromobenzene	2	0.027	ug/L
8260B	5030B	Water	Bromochloromethane	0.5	0.091	ug/L
8260B	5030B	Water	Bromodichloromethane	0.5	0.036	ug/L

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8260B	5030B	Water	Bromoform	0.5	0.080	ug/L
8260B	5030B	Water	Bromomethane	0.5	0.072	ug/L
8260B	5030B	Water	Carbon Disulfide	0.5	0.045	ug/L
8260B	5030B	Water	Carbon Tetrachloride	0.5	0.068	ug/L
8260B	5030B	Water	Chlorobenzene	0.5	0.045	ug/L
8260B	5030B	Water	Chloroethane	0.5	0.13	ug/L
8260B	5030B	Water	Chloroform	0.5	0.042	ug/L
8260B	5030B	Water	Chloromethane	0.5	0.053	ug/L
8260B	5030B	Water	Chloroprene	10	0.15	ug/L
8260B	5030B	Water	cis-1,2-Dichloroethene	0.5	0.045	ug/L
8260B	5030B	Water	cis-1,3-Dichloropropene	0.5	0.038	ug/L
8260B	5030B	Water	cis-1,4-Dichloro-2-butene	10	0.84	ug/L
8260B	5030B	Water	Cyclohexane	1	0.18	ug/L
8260B	5030B	Water	Dibromochloromethane	0.5	0.057	ug/L
8260B	5030B	Water	Dibromomethane	0.5	0.089	ug/L
8260B	5030B	Water	Dichlorodifluoromethane	0.5	0.083	ug/L
8260B	5030B	Water	Dichlorofluoromethane (CFC 21)	0.5	0.053	ug/L
8260B	5030B	Water	Diisopropyl Ether	2	0.046	ug/L
8260B	5030B	Water	Ethyl Acetate	5	0.81	ug/L
8260B	5030B	Water	Ethyl Ether	1	0.069	ug/L
8260B	5030B	Water	Ethyl Methacrylate	5	0.11	ug/L
8260B	5030B	Water	Ethylbenzene	0.5	0.042	ug/L
8260B	5030B	Water	Ethylene Oxide	10	1.1	ug/L
8260B	5030B	Water	Hexachlorobutadiene	2	0.19	ug/L
8260B	5030B	Water	Iodomethane	5	0.27	ug/L
8260B	5030B	Water	Isobutyl Alcohol	100	12	ug/L
8260B	5030B	Water	Isopropylbenzene	2	0.031	ug/L
8260B	5030B	Water	m,p-Xylenes	0.5	0.078	ug/L
8260B	5030B	Water	Methacrylonitrile	5	0.25	ug/L
8260B	5030B	Water	Methyl Acetate	1	0.26	ug/L
8260B	5030B	Water	Methyl Methacrylate	5	0.18	ug/L
8260B	5030B	Water	Methyl tert-Butyl Ether	0.5	0.070	ug/L

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8260B	5030B	Water	Methylcyclohexane	1	0.18	ug/L
8260B	5030B	Water	Methylene Chloride	2	0.23	ug/L
8260B	5030B	Water	Naphthalene	2	0.10	ug/L
8260B	5030B	Water	n-Butylbenzene	2	0.056	ug/L
8260B	5030B	Water	n-Hexane	1	0.29	ug/L
8260B	5030B	Water	n-Octane	5	0.33	ug/L
8260B	5030B	Water	n-Propylbenzene	2	0.037	ug/L
8260B	5030B	Water	o-Xylene	0.5	0.037	ug/L
8260B	5030B	Water	Propionitrile	5	1.1	ug/L
8260B	5030B	Water	sec-Butylbenzene	2	0.036	ug/L
8260B	5030B	Water	Styrene	0.5	0.039	ug/L
8260B	5030B	Water	tert-Amyl Methyl Ether	2	0.078	ug/L
8260B	5030B	Water	tert-Butyl Alcohol	20	1.2	ug/L
8260B	5030B	Water	tert-Butyl Ethyl Ether	2	0.027	ug/L
8260B	5030B	Water	tert-Butylbenzene	2	0.038	ug/L
8260B	5030B	Water	Tetrachloroethene (PCE)	0.5	0.077	ug/L
8260B	5030B	Water	Tetrahydrofuran	5	0.89	ug/L
8260B	5030B	Water	Toluene	0.5	0.048	ug/L
8260B	5030B	Water	trans-1,2-Dichloroethene	0.5	0.048	ug/L
8260B	5030B	Water	trans-1,3-Dichloropropene	0.5	0.041	ug/L
8260B	5030B	Water	trans-1,4-Dichloro-2-butene	10	0.20	ug/L
8260B	5030B	Water	Trichloroethene (TCE)	0.5	0.061	ug/L
8260B	5030B	Water	Trichlorofluoromethane	0.5	0.086	ug/L
8260B	5030B	Water	Trichlorotrifluoroethane	0.5	0.079	ug/L
8260B	5030B	Water	Vinyl Acetate	5	0.91	ug/L
8260B	5030B	Water	Vinyl Chloride	0.5	0.071	ug/L
AK 101	Method	Soil	Gasoline Range Petroleum Hydrocarbons	20	1.5	mg/kg
8015B	5035/5030B	Soil	Gasoline Range Petroleum Hydrocarbons	5	1.3	mg/kg
NWTPH-Gx	5035/5030B	Soil	Gasoline Range Petroleum Hydrocarbons	5	1.5	mg/kg
AK 101	Method	Water	Gasoline Range Petroleum Hydrocarbons	100	13	ug/L
8015B	5030B	Water	Gasoline Range Petroleum Hydrocarbons	50	13	ug/L
NWTPH-Gx	5030B	Water	Gasoline Range Petroleum Hydrocarbons	250	13	ug/L

VOLATILE ORGANIC COMPOUNDS (VOCs) ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**

a Method Detection Limits are subject to change as new MDL studies are completed.

ORGANIC COMPOUNDS IN DRINKING WATER ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
504.1	Method	DW	1,2,3-Trichloropropane	0.05	0.021	ug/L
504.1	Method	DW	1,2-Dibromo-3-chloropropane (DBCP)	0.01	0.0028	ug/L
504.1	Method	DW	Ethylene Dibromide (EDB)	0.01	0.0041	ug/L
508.1	Method	DW	4,4'-DDD	0.01	0.00076	ug/L
508.1	Method	DW	4,4'-DDE	0.01	0.00082	ug/L
508.1	Method	DW	4,4'-DDT	0.01	0.00088	ug/L
508.1	Method	DW	Aldrin	0.01	0.0012	ug/L
508.1	Method	DW	alpha-BHC	0.01	0.00052	ug/L
508.1	Method	DW	alpha-Chlordane	0.01	0.0014	ug/L
508.1	Method	DW	beta-BHC	0.01	0.00084	ug/L
508.1	Method	DW	Chlordane	0.1	0.01	ug/L
508.1	Method	DW	delta-BHC	0.01	0.001	ug/L
508.1	Method	DW	Dieldrin	0.01	0.0027	ug/L
508.1	Method	DW	Endosulfan I	0.01	0.0014	ug/L
508.1	Method	DW	Endosulfan II	0.01	0.0013	ug/L
508.1	Method	DW	Endosulfan Sulfate	0.01	0.00085	ug/L
508.1	Method	DW	Endrin	0.01	0.00075	ug/L
508.1	Method	DW	Endrin Aldehyde	0.01	0.0014	ug/L
508.1	Method	DW	gamma-BHC (Lindane)	0.01	0.00093	ug/L
508.1	Method	DW	gamma-Chlordane	0.01	0.0011	ug/L
508.1	Method	DW	Heptachlor	0.01	0.00094	ug/L
508.1	Method	DW	Heptachlor Epoxide	0.01	0.0021	ug/L
508.1	Method	DW	Heptachlor Epoxide (Isomer A)	0.01	0.0017	ug/L
508.1	Method	DW	Methoxychlor	0.01	0.0012	ug/L
508.1	Method	DW	Toxaphene	0.1	0.026	ug/L
508.1	Method	DW	Aroclor 1016	0.05	0.026	ug/L
508.1	Method	DW	Aroclor 1221	0.1	0.033	ug/L
508.1	Method	DW	Aroclor 1232	0.1	0.016	ug/L
508.1	Method	DW	Aroclor 1242	0.1	0.018	ug/L
508.1	Method	DW	Aroclor 1248	0.1	0.03	ug/L
508.1	Method	DW	Aroclor 1254	0.1	0.019	ug/L
508.1	Method	DW	Aroclor 1260	0.1	0.034	ug/L

ORGANIC COMPOUNDS IN DRINKING WATER ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
515.4	Method	DW	2,4,5-T	0.2	0.0079	ug/L
515.4	Method	DW	2,4,5-TP (Silvex)	0.05	0.0076	ug/L
515.4	Method	DW	2,4-D	0.1	0.044	ug/L
515.4	Method	DW	2,4-DB	0.4	0.033	ug/L
515.4	Method	DW	3,5-Dichlorobenzoic Acid	0.2	0.02	ug/L
515.4	Method	DW	4-Nitrophenol	0.4	0.08	ug/L
515.4	Method	DW	Acifluorfen	0.2	0.014	ug/L
515.4	Method	DW	Bentazon	0.4	0.041	ug/L
515.4	Method	DW	Chloramben	0.3	0.016	ug/L
515.4	Method	DW	Dacthal Diacid	0.2	0.029	ug/L
515.4	Method	DW	Dalapon	1	0.099	ug/L
515.4	Method	DW	Dicamba	0.2	0.023	ug/L
515.4	Method	DW	Dichlorprop	0.4	0.029	ug/L
515.4	Method	DW	Dinoseb	0.2	0.026	ug/L
515.4	Method	DW	Pentachlorophenol	0.04	0.0093	ug/L
515.4	Method	DW	Picloram	0.1	0.025	ug/L
524.2	Method	DW	1,1,1,2-Tetrachloroethane	.50	0.031	ug/L
524.2	Method	DW	1,1,1-Trichloroethane	.50	0.057	ug/L
524.2	Method	DW	1,1,2,2-Tetrachloroethane	.50	0.031	ug/L
524.2	Method	DW	1,1,2-Trichloroethane	.50	0.079	ug/L
524.2	Method	DW	1,1-Dichloroethane	.50	0.041	ug/L
524.2	Method	DW	1,1-Dichloroethene	.50	0.066	ug/L
524.2	Method	DW	1,1-Dichloropropanone	10	1.6	ug/L
524.2	Method	DW	1,1-Dichloropropene	.50	0.038	ug/L
524.2	Method	DW	1,2,3-Trichlorobenzene	.50	0.031	ug/L
524.2	Method	DW	1,2,3-Trichloropropane	.50	0.15	ug/L
524.2	Method	DW	1,2,4-Trichlorobenzene	.50	0.025	ug/L
524.2	Method	DW	1,2,4-Trimethylbenzene	.50	0.017	ug/L
524.2	Method	DW	1,2-Dibromo-3-chloropropane (DBCP)	.50	0.12	ug/L
524.2	Method	DW	1,2-Dibromoethane (EDB)	.50	0.083	ug/L
524.2	Method	DW	1,2-Dichlorobenzene	.50	0.015	ug/L

ORGANIC COMPOUNDS IN DRINKING WATER ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
524.2	Method	DW	1,2-Dichloroethane	.50	0.028	ug/L
524.2	Method	DW	1,2-Dichloropropane	.50	0.044	ug/L
524.2	Method	DW	1,3,5-Trichlorobenzene	.50	0.5	ug/L
524.2	Method	DW	1,3,5-Trimethylbenzene	.50	0.015	ug/L
524.2	Method	DW	1,3-Dichlorobenzene	.50	0.024	ug/L
524.2	Method	DW	1,3-Dichloropropane	.50	0.031	ug/L
524.2	Method	DW	1,4-Dichlorobenzene	.50	0.036	ug/L
524.2	Method	DW	2,2-Dichloropropane	.50	0.047	ug/L
524.2	Method	DW	2-Butanone (MEK)	20	1.3	ug/L
524.2	Method	DW	2-Chlorotoluene	.50	0.028	ug/L
524.2	Method	DW	2-Hexanone	20	0.63	ug/L
524.2	Method	DW	2-Nitropropane	2.0	0.59	ug/L
524.2	Method	DW	3-Chloro-1-propene	2.0	0.33	ug/L
524.2	Method	DW	4-Chlorotoluene	.50	0.028	ug/L
524.2	Method	DW	4-Methyl-2-pentanone (MIBK)	20	1.3	ug/L
524.2	Method	DW	Acetone	20	1.5	ug/L
524.2	Method	DW	Acrylonitrile	2.0	0.2	ug/L
524.2	Method	DW	Benzene	.50	0.039	ug/L
524.2	Method	DW	Bromobenzene	.50	0.04	ug/L
524.2	Method	DW	Bromochloromethane	.50	0.035	ug/L
524.2	Method	DW	Bromodichloromethane	.50	0.062	ug/L
524.2	Method	DW	Bromoform	.50	0.048	ug/L
524.2	Method	DW	Bromomethane	.50	0.066	ug/L
524.2	Method	DW	Carbon Disulfide	.50	0.054	ug/L
524.2	Method	DW	Carbon Tetrachloride	.50	0.074	ug/L
524.2	Method	DW	Chlorobenzene	.50	0.031	ug/L
524.2	Method	DW	Chloroethane	.50	0.22	ug/L
524.2	Method	DW	Chloroform	.50	0.017	ug/L
524.2	Method	DW	Chloromethane	.50	0.066	ug/L
524.2	Method	DW	cis-1,2-Dichloroethene	.50	0.041	ug/L

ORGANIC COMPOUNDS IN DRINKING WATER ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
524.2	Method	DW	cis-1,3-Dichloropropene	.50	0.042	ug/L
524.2	Method	DW	cis-1,4-Dichloro-2-butene	2.0	0.41	ug/L
524.2	Method	DW	Dibromochloromethane	.50	0.03	ug/L
524.2	Method	DW	Dibromomethane	.50	0.076	ug/L
524.2	Method	DW	Dichlorodifluoromethane	.50	0.066	ug/L
524.2	Method	DW	Diisopropyl Ether	.50	0.025	ug/L
524.2	Method	DW	Ethyl Ether	1.0	0.047	ug/L
524.2	Method	DW	Ethyl Methacrylate	1.0	0.085	ug/L
524.2	Method	DW	Ethylbenzene	.50	0.024	ug/L
524.2	Method	DW	Hexachlorobutadiene	.50	0.036	ug/L
524.2	Method	DW	Iodomethane	1.0	0.054	ug/L
524.2	Method	DW	Isopropylbenzene	.50	0.015	ug/L
524.2	Method	DW	m,p-Xylenes	.50	0.054	ug/L
524.2	Method	DW	Methacrylonitrile	1.0	0.21	ug/L
524.2	Method	DW	Methyl Methacrylate	1.0	0.21	ug/L
524.2	Method	DW	Methyl tert-Butyl Ether	.50	0.044	ug/L
524.2	Method	DW	Methylene Chloride	.50	0.16	ug/L
524.2	Method	DW	Naphthalene	.50	0.028	ug/L
524.2	Method	DW	n-Butylbenzene	.50	0.026	ug/L
524.2	Method	DW	n-Propylbenzene	.50	0.031	ug/L
524.2	Method	DW	*o-Xylene*	.50	0.036	ug/L
524.2	Method	DW	p-Isopropyltoluene	.50	0.028	ug/L
524.2	Method	DW	Propionitrile	20	7.6	ug/L
524.2	Method	DW	sec-Butylbenzene	.50	0.018	ug/L
524.2	Method	DW	Styrene	.50	0.051	ug/L
524.2	Method	DW	tert-Amyl Methyl Ether	.50	0.05	ug/L
524.2	Method	DW	tert-Butyl Alcohol	20	1.6	ug/L
524.2	Method	DW	tert-Butyl Ethyl Ether	.50	0.024	ug/L
524.2	Method	DW	tert-Butylbenzene	.50	0.015	ug/L
524.2	Method	DW	Tetrachloroethene	.50	0.064	ug/L
524.2	Method	DW	Tetrahydrofuran	20	5.4	ug/L

ORGANIC COMPOUNDS IN DRINKING WATER ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
524.2	Method	DW	Toluene	.50	0.041	ug/L
524.2	Method	DW	trans-1,2-Dichloroethene	.50	0.047	ug/L
524.2	Method	DW	trans-1,3-Dichloropropene	.50	0.042	ug/L
524.2	Method	DW	trans-1,4-Dichloro-2-butene	1.0	0.51	ug/L
524.2	Method	DW	Trichloroethene	.50	0.075	ug/L
524.2	Method	DW	Trichlorofluoromethane	.50	0.072	ug/L
524.2	Method	DW	Trichlorotrifluoroethane	.50	0.067	ug/L
524.2	Method	DW	Vinyl Chloride	.50	0.062	ug/L
525.2	Method	DW	2,4-Dinitrotoluene	0.5	0.0099	ug/L
525.2	Method	DW	2,6-Dinitrotoluene	0.1	0.028	ug/L
525.2	Method	DW	4,4'-DDD	0.05	0.007	ug/L
525.2	Method	DW	4,4'-DDE	0.05	0.0087	ug/L
525.2	Method	DW	4,4'-DDT	0.050	0.0052	ug/L
525.2	Method	DW	Acenaphthene	0.050	0.0064	ug/L
525.2	Method	DW	Acenaphthylene	0.050	0.0065	ug/L
525.2	Method	DW	Acetochlor	1	0.0085	ug/L
525.2	Method	DW	Alachlor	0.072	0.0071	ug/L
525.2	Method	DW	Aldrin	0.094	0.0086	ug/L
525.2	Method	DW	alpha-BHC	0.05	0.0066	ug/L
525.2	Method	DW	alpha-Chlordane	0.076	0.0095	ug/L
525.2	Method	DW	Ametryn	0.2	0.2	ug/L
525.2	Method	DW	Anthracene	0.068	0.0074	ug/L
525.2	Method	DW	Atraton	0.2	0.2	ug/L
525.2	Method	DW	Atrazine	0.10	0.014	ug/L
525.2	Method	DW	Benz(a)anthracene	0.2	0.0082	ug/L
525.2	Method	DW	Benzo(a)pyrene	0.02	0.0093	ug/L
525.2	Method	DW	Benzo(b)fluoranthene	0.2	0.0044	ug/L
525.2	Method	DW	Benzo(g,h,i)perylene	0.2	0.019	ug/L
525.2	Method	DW	Benzo(k)fluoranthene	0.2	0.013	ug/L
525.2	Method	DW	Benzyl butyl phthalate	0.5	0.013	ug/L
525.2	Method	DW	beta-BHC	0.054	0.011	ug/L
525.2	Method	DW	Bis(2-ethylhexyl) Adipate	0.6	0.013	ug/L

ORGANIC COMPOUNDS IN DRINKING WATER ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
525.2	Method	DW	Bis(2-ethylhexyl) Phthalate	0.6	0.04	ug/L
525.2	Method	DW	Bromacil	0.2	0.013	ug/L
525.2	Method	DW	Butachlor	0.052	0.0032	ug/L
525.2	Method	DW	Butylate	0.05	0.0051	ug/L
525.2	Method	DW	Chrysene	0.2	0.0043	ug/L
525.2	Method	DW	cis-Permethrin	0.2	0.0039	ug/L
525.2	Method	DW	Cyanazine	0.1	0.012	ug/L
525.2	Method	DW	Dacthal	0.05	0.014	ug/L
525.2	Method	DW	delta-BHC	0.05	0.0092	ug/L
525.2	Method	DW	Diazinon	0.2	0.0099	ug/L
525.2	Method	DW	Dibenz(a,h)anthracene	0.2	0.015	ug/L
525.2	Method	DW	Dieldrin	0.2	0.013	ug/L
525.2	Method	DW	Diethyl Phthalate	0.5	0.015	ug/L
525.2	Method	DW	Dimethyl Phthalate	0.5	0.0073	ug/L
525.2	Method	DW	Di-n-butyl Phthalate	0.6	0.05	ug/L
525.2	Method	DW	Di-n-octyl Phthalate	0.2	0.0062	ug/L
525.2	Method	DW	Endosulfan I	0.2	0.0088	ug/L
525.2	Method	DW	Endosulfan II	0.2	0.008	ug/L
525.2	Method	DW	Endosulfan Sulfate	0.076	0.013	ug/L
525.2	Method	DW	Endrin	0.054	0.0096	ug/L
525.2	Method	DW	Endrin Aldehyde	0.2	0.0073	ug/L
525.2	Method	DW	Endrin Ketone	0.2	0.013	ug/L
525.2	Method	DW	EPTC	0.052	0.01	ug/L
525.2	Method	DW	Fluoranthene	0.1	0.0061	ug/L
525.2	Method	DW	Fluorene	0.05	0.0078	ug/L
525.2	Method	DW	gamma-BHC (Lindane)	0.05	0.0083	ug/L
525.2	Method	DW	gamma-Chlordane	0.05	0.0075	ug/L
525.2	Method	DW	Heptachlor	0.080	0.01	ug/L
525.2	Method	DW	Heptachlor Epoxide	0.05	0.0076	ug/L
525.2	Method	DW	Hexachlorobenzene	0.1	0.0069	ug/L
525.2	Method	DW	Hexachlorocyclopentadiene	0.1	0.012	ug/L
525.2	Method	DW	Indeno(1,2,3-cd)pyrene	0.2	0.017	ug/L

ORGANIC COMPOUNDS IN DRINKING WATER ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
525.2	Method	DW	Isophorone	0.05	0.0079	ug/L
525.2	Method	DW	Malathion	0.2	0.0077	ug/L
525.2	Method	DW	Methoxychlor	0.05	0.0035	ug/L
525.2	Method	DW	Metolachlor	0.090	0.0032	ug/L
525.2	Method	DW	Metribuzin	0.2	0.0084	ug/L
525.2	Method	DW	Molinate	0.05	0.0057	ug/L
525.2	Method	DW	Parathion	0.2	0.011	ug/L
525.2	Method	DW	Pebulate	0.05	0.0085	ug/L
525.2	Method	DW	Phenanthrene	0.05	0.0032	ug/L
525.2	Method	DW	Prometon	0.05	0.0045	ug/L
525.2	Method	DW	Prometryn	0.1	0.0033	ug/L
525.2	Method	DW	Pronamide	0.2	0.0088	ug/L
525.2	Method	DW	Propachlor	0.076	0.0067	ug/L
525.2	Method	DW	Propazine	0.05	0.015	ug/L
525.2	Method	DW	Pyrene	0.05	0.0065	ug/L
525.2	Method	DW	Simazine	0.05	0.012	ug/L
525.2	Method	DW	Simetryn	0.1	0.1	ug/L
525.2	Method	DW	Terbacil	0.2	0.011	ug/L
525.2	Method	DW	trans-Nonachlor	0.090	0.0071	ug/L
525.2	Method	DW	trans-Permethrin	0.2	0.0099	ug/L
525.2	Method	DW	Trifluralin	0.1	0.0036	ug/L
531.1	Method	DW	3-Hydroxycarbofuran	0.5	0.27	ug/L
531.1	Method	DW	Aldicarb	0.5	0.12	ug/L
531.1	Method	DW	Aldicarb Sulfone	0.5	0.21	ug/L
531.1	Method	DW	Aldicarb Sulfoxide	0.5	0.18	ug/L
531.1	Method	DW	Carbaryl	0.5	0.18	ug/L
531.1	Method	DW	Carbofuran	0.5	0.26	ug/L
531.1	Method	DW	Methiocarb	0.5	0.31	ug/L
531.1	Method	DW	Methomyl	0.5	0.19	ug/L
531.1	Method	DW	Oxamyl	0.5	0.12	ug/L
531.1	Method	DW	Propoxur	0.5	0.28	ug/L
547	Method	DW	Glyphosate	6	1.7	ug/L

ORGANIC COMPOUNDS IN DRINKING WATER ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
548.1	Method	DW	Endothall	5	1.2	ug/L
549.2	Method	DW	Diquat	0.4	0.26	ug/L
549.2	Method	DW	Paraquat	0.8	0.76	ug/L
552.2	Method	DW	Bromoacetic Acid	1.0	0.22	ug/L
552.2	Method	DW	Bromochloroacetic Acid	1.0	0.17	ug/L
552.2	Method	DW	Chloroacetic Acid	2.0	0.28	ug/L
552.2	Method	DW	Dibromoacetic Acid	1.0	0.19	ug/L
552.2	Method	DW	Dichloroacetic Acid	1.0	0.17	ug/L
552.2	Method	DW	Trichloroacetic Acid	1.0	0.27	ug/L

a Method Detection Limits are subject to change as new MDL studies are completed.

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**
ASTM D4129-82M	Method	Sediment	Total Organic Carbon	0.05	0.04	%
9030M	Method	Sediment	Sulfides	0.5	0.05	mg/kg
PSEP	PSEP	Sediment	Sulfides	1	0.6	mg/kg
Lab SOP	Lab SOP	Sediment	Acid Volatile Sulfides	0.5	0.09	mg/kg
PSEP	NA	Sediment	Particle Size	NA	NA	
6010B - ICP	AVS-SEM	Sediment	Cadmium	0.2	-	mg/kg
6010B - ICP	AVS-SEM	Sediment	Copper	0.4	-	mg/kg
6010B - ICP	AVS-SEM	Sediment	Lead	3	-	mg/kg
6010B - ICP	AVS-SEM	Sediment	Nickel	0.5	-	mg/kg
6010B - ICP	AVS-SEM	Sediment	Zinc	0.4	-	mg/kg
7470A	AVS-SEM	Sediment	Mercury	0.01	-	mg/kg
200.8 - ICP/MS	3050B	Sediment	Aluminum	2	0.5	mg/kg
200.8 - ICP/MS	3050B	Sediment	Antimony	0.05	0.05	mg/kg
200.8 - ICP/MS	3050B	Sediment	Arsenic	0.5	0.2	mg/kg
200.8 - ICP/MS	3050B	Sediment	Barium	0.05	0.02	mg/kg
200.8 - ICP/MS	3050B	Sediment	Beryllium	0.02	0.005	mg/kg
200.8 - ICP/MS	3050B	Sediment	Cadmium	0.02	0.009	mg/kg
6010B - ICP	3050B	Sediment	Chromium	2	0.7	mg/kg
200.8 - ICP/MS	3050B	Sediment	Cobalt	0.02	0.004	mg/kg
200.8 - ICP/MS	3050B	Sediment	Copper	0.1	0.09	mg/kg
200.8 - ICP/MS	3050B	Sediment	Lead	0.05	0.05	mg/kg
200.8 - ICP/MS	3050B	Sediment	Manganese	0.05	0.02	mg/kg
7471A- CVAA	Method	Sediment	Mercury	0.02	0.002	mg/kg
200.8 - ICP/MS	3050B	Sediment	Molybdenum	0.05	0.04	mg/kg
200.8 - ICP/MS	3050B	Sediment	Nickel	0.2	0.03	mg/kg
7740 - GFAA	3050B	Sediment	Selenium	1	0.06	mg/kg
200.8 - ICP/MS	3050B	Sediment	Silver	0.02	0.02	mg/kg
200.8 - ICP/MS	3050B	Sediment	Thallium	0.02	0.005	mg/kg
6010B - ICP	3050B	Sediment	Vanadium	2	0.7	mg/kg
200.8 - ICP/MS	3050B	Sediment	Zinc	0.5	0.5	mg/kg
200.8 - ICP/MS	Reductive Precip.	Seawater	Arsenic	0.5	0.02	ug/L
200.8 - ICP/MS	Reductive Precip.	Seawater	Beryllium	0.02	0.0007	ug/L
200.8 - ICP/MS	Reductive Precip.	Seawater	Cadmium	0.02	0.006	ug/L

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**
200.8 - ICP/MS	Reductive Precip.	Seawater	Chromium	0.2	0.03	ug/L
200.8 - ICP/MS	Reductive Precip.	Seawater	Cobalt	0.02	0.001	ug/L
200.8 - ICP/MS	Reductive Precip.	Seawater	Copper	0.1	0.03	ug/L
200.8 - ICP/MS	Reductive Precip.	Seawater	Lead	0.02	0.003	ug/L
200.8 - ICP/MS	Reductive Precip.	Seawater	Nickel	0.2	0.03	ug/L
200.8 - ICP/MS	Reductive Precip.	Seawater	Silver	0.02	0.004	ug/L
200.8 - ICP/MS	Reductive Precip.	Seawater	Thallium	0.02	0.0009	ug/L
200.8 - ICP/MS	Reductive Precip.	Seawater	Zinc	0.5	0.04	ug/L
7062	3010A/BRAA	Seawater	Arsenic	1	0.2	ug/L
7742	3010A/BRAA	Seawater	Selenium	1	0.2	ug/L
200.8 - ICP/MS	3050B/PSEP	Tissue	Aluminum	2	0.2	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Antimony	0.05	0.02	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Arsenic	0.5	0.08	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Barium	0.05	0.03	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Beryllium	0.02	0.007	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Cadmium	0.02	0.02	mg/kg
6010B - ICP	3050B/PSEP	Tissue	Chromium	1	0.4	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Cobalt	0.02	0.003	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Copper	0.1	0.08	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Lead	0.02	0.008	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Manganese	0.05	0.006	mg/kg
7471A- CVAA	Method	Tissue	Mercury	0.02	0.004	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Molybdenum	0.05	0.03	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Nickel	0.2	0.04	mg/kg
7740 - GFAA	3050B/PSEP	Tissue	Selenium	1	0.5	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Silver	0.02	0.008	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Thallium	0.02	0.005	mg/kg
6010B - ICP	3050B/PSEP	Tissue	Vanadium	1	0.5	mg/kg
200.8 - ICP/MS	3050B/PSEP	Tissue	Zinc	0.5	0.09	mg/kg
7062	PSEP	Tissue	Arsenic	0.1	0.04	mg/kg
7742	PSEP	Tissue	Selenium	0.1	0.02	mg/kg
8081A	3540C	Sed.-Low	2,4'-DDD	1.0	0.21	ug/kg
8081A	3540C	Sed.-Low	2,4'-DDE	1.0	0.23	ug/kg

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**
8081A	3540C	Sed.-Low	2,4'-DDT	1.0	0.14	ug/kg
8081A	3540C	Sed.-Low	4,4'-DDD	1.0	0.12	ug/kg
8081A	3540C	Sed.-Low	4,4'-DDE	1.0	0.10	ug/kg
8081A	3540C	Sed.-Low	4,4'-DDT	1.0	0.064	ug/kg
8081A	3540C	Sed.-Low	Aldrin	1.0	0.15	ug/kg
8081A	3540C	Sed.-Low	alpha-BHC	1.0	0.26	ug/kg
8081A	3540C	Sed.-Low	alpha-Chlordane	1.0	0.23	ug/kg
8081A	3540C	Sed.-Low	beta-BHC	1.0	0.30	ug/kg
8081A	3540C	Sed.-Low	Chlordane	10	8.1	ug/kg
8081A	3540C	Sed.-Low	Chlorpyrifos	1.0	0.25	ug/kg
8081A	3540C	Sed.-Low	cis-Nonachlor	1.0	0.071	ug/kg
8081A	3540C	Sed.-Low	delta-BHC	1.0	0.055	ug/kg
8081A	3540C	Sed.-Low	Dieldrin	1.0	0.29	ug/kg
8081A	3540C	Sed.-Low	Endosulfan I	1.0	0.17	ug/kg
8081A	3540C	Sed.-Low	Endosulfan II	1.0	0.19	ug/kg
8081A	3540C	Sed.-Low	Endosulfan Sulfate	1.0	0.079	ug/kg
8081A	3540C	Sed.-Low	Endrin	1.0	0.20	ug/kg
8081A	3540C	Sed.-Low	Endrin Aldehyde	1.0	0.053	ug/kg
8081A	3540C	Sed.-Low	Endrin Ketone	1.0	0.082	ug/kg
8081A	3540C	Sed.-Low	gamma-BHC (Lindane)	1.0	0.15	ug/kg
8081A	3540C	Sed.-Low	gamma-Chlordane	1.0	0.064	ug/kg
8081A	3540C	Sed.-Low	Heptachlor	1.0	0.080	ug/kg
8081A	3540C	Sed.-Low	Heptachlor Epoxide	1.0	0.13	ug/kg
8081A	3540C	Sed.-Low	Hexachlorobenzene	1.0	0.079	ug/kg
8081A	3540C	Sed.-Low	Hexachlorobutadiene	1.0	0.13	ug/kg
8081A	3540C	Sed.-Low	Hexachlorocyclopentadiene	1.0	0.39	ug/kg
8081A	3540C	Sed.-Low	Hexachloroethane	1.0	0.15	ug/kg
8081A	3540C	Sed.-Low	Isodrin	1.0	0.097	ug/kg
8081A	3540C	Sed.-Low	Methoxychlor	1.0	0.10	ug/kg
8081A	3540C	Sed.-Low	Mirex	1.0	0.063	ug/kg
8081A	3540C	Sed.-Low	Oxychlordane	1.0	0.058	ug/kg
8081A	3540C	Sed.-Low	Toxaphene	50	9.2	ug/kg
8081A	3540C	Sed.-Low	trans-Nonachlor	1.0	0.066	ug/kg
8081A	3540C	Tissue	2,4'-DDD	1.0	0.38	ug/kg
8081A	3540C	Tissue	2,4'-DDE	1.0	0.21	ug/kg

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**
8081A	3540C	Tissue	2,4'-DDT	1.0	0.21	ug/kg
8081A	3540C	Tissue	4,4'-DDD	1.0	0.11	ug/kg
8081A	3540C	Tissue	4,4'-DDE	1.0	0.16	ug/kg
8081A	3540C	Tissue	4,4'-DDT	1.0	0.43	ug/kg
8081A	3540C	Tissue	Aldrin	1.0	0.23	ug/kg
8081A	3540C	Tissue	alpha-BHC	1.0	0.27	ug/kg
8081A	3540C	Tissue	alpha-Chlordane	1.0	0.15	ug/kg
8081A	3540C	Tissue	beta-BHC	1.0	0.49	ug/kg
8081A	3540C	Tissue	Chlordane	10	4.1	ug/kg
8081A	3540C	Tissue	Chlorpyrifos	1.0	0.25	ug/kg
8081A	3540C	Tissue	cis-Nonachlor	1.0	0.19	ug/kg
8081A	3540C	Tissue	delta-BHC	1.0	0.16	ug/kg
8081A	3540C	Tissue	Dieldrin	1.0	0.25	ug/kg
8081A	3540C	Tissue	Endosulfan I	1.0	0.12	ug/kg
8081A	3540C	Tissue	Endosulfan II	1.0	0.22	ug/kg
8081A	3540C	Tissue	Endosulfan Sulfate	1.0	0.13	ug/kg
8081A	3540C	Tissue	Endrin	1.0	0.22	ug/kg
8081A	3540C	Tissue	Endrin Aldehyde	1.0	0.25	ug/kg
8081A	3540C	Tissue	Endrin Ketone	1.0	0.28	ug/kg
8081A	3540C	Tissue	gamma-BHC (Lindane)	1.0	0.24	ug/kg
8081A	3540C	Tissue	gamma-Chlordane	1.0	0.26	ug/kg
8081A	3540C	Tissue	Heptachlor	1.0	0.66	ug/kg
8081A	3540C	Tissue	Heptachlor Epoxide	1.0	0.38	ug/kg
8081A	3540C	Tissue	Hexachlorobenzene	1.0	0.31	ug/kg
8081A	3540C	Tissue	Hexachlorobutadiene	1.0	0.13	ug/kg
8081A	3540C	Tissue	Hexachloroethane	1.0	0.15	ug/kg
8081A	3540C	Tissue	Isodrin	1.0	0.25	ug/kg
8081A	3540C	Tissue	Methoxychlor	1.0	1.0	ug/kg
8081A	3540C	Tissue	Mirex	1.0	0.40	ug/kg
8081A	3540C	Tissue	Oxychlordane	1.0	0.19	ug/kg
8081A	3540C	Tissue	Toxaphene	50	21	ug/kg
8081A	3540C	Tissue	trans-Nonachlor	1.0	0.14	ug/kg
8082 Aroclors	3541	Sed.-Low	Aroclor 1016	10	1.7	ug/kg
8082 Aroclors	3541	Sed.-Low	Aroclor 1221	20	1.7	ug/kg
8082 Aroclors	3541	Sed.-Low	Aroclor 1232	10	1.7	ug/kg

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**
8082 Aroclors	3541	Sed.-Low	Aroclor 1242	10	1.7	ug/kg
8082 Aroclors	3541	Sed.-Low	Aroclor 1248	10	1.7	ug/kg
8082 Aroclors	3541	Sed.-Low	Aroclor 1254	10	1.7	ug/kg
8082 Aroclors	3541	Sed.-Low	Aroclor 1260	10	1.7	ug/kg
8082 Aroclors	3541	Sed.-Low	Aroclor 1262	10	1.7	ug/kg
8082 Aroclors	3541	Sed.-Low	Aroclor 1268	10	1.7	ug/kg
8082 Aroclors	3540C	Tissue	Aroclor 1016	10	2.4	ug/kg
8082 Aroclors	3540C	Tissue	Aroclor 1221	20	2.6	ug/kg
8082 Aroclors	3540C	Tissue	Aroclor 1232	10	2.3	ug/kg
8082 Aroclors	3540C	Tissue	Aroclor 1242	10	2.2	ug/kg
8082 Aroclors	3540C	Tissue	Aroclor 1248	10	0.51	ug/kg
8082 Aroclors	3540C	Tissue	Aroclor 1254	10	1.8	ug/kg
8082 Aroclors	3540C	Tissue	Aroclor 1260	10	1.9	ug/kg
8082 Aroclors	3540C	Tissue	Aroclor 1262	10	2.5	ug/kg
8082 Aroclors	3540C	Tissue	Aroclor 1268	10	2.0	ug/kg
8082 Congeners	3540C	Soil	PCB1 (2-Chlorobiphenyl)	5.0		ug/kg
8082 Congeners	3540C	Soil	PCB101 (2,2',4,5,5'-Pentachlorobiphenyl)	0.50	0.090	ug/kg
8082 Congeners	3540C	Soil	PCB105 (2,3,3',4,4'-Pentachlorobiphenyl)	0.50	0.13	ug/kg
8082 Congeners	3540C	Soil	PCB110 (2,3,3',4',6-Pentachlorobiphenyl)	0.50	0.088	ug/kg
8082 Congeners	3540C	Soil	PCB114 (2,3,4,4',5-Pentachlorobiphenyl)	0.50	0.077	ug/kg
8082 Congeners	3540C	Soil	PCB118 (2,3',4,4',5-Pentachlorobiphenyl)	0.50	0.13	ug/kg
8082 Congeners	3540C	Soil	PCB119 (2,3',4,4',6-Pentachlorobiphenyl)	0.50	0.17	ug/kg
8082 Congeners	3540C	Soil	PCB123 (2',3,4,4',5-Pentachlorobiphenyl)	0.50	0.13	ug/kg
8082 Congeners	3540C	Soil	PCB126 (3,3',4,4',5-Pentachlorobiphenyl)	0.50	0.35	ug/kg
8082 Congeners	3540C	Soil	PCB128 (2,2',3,3',4,4'-Hexachlorobiphenyl)	0.50	0.14	ug/kg
8082 Congeners	3540C	Soil	PCB132 (2,2',3,3',4,6'-Hexachlorobiphenyl)	0.50	0.089	ug/kg
8082 Congeners	3540C	Soil	PCB138 (2,2',3,4,4',5'-Hexachlorobiphenyl)	0.50	0.093	ug/kg
8082 Congeners	3540C	Soil	PCB141 (2,2',3,4,5,5'-Hexachlorobiphenyl)	0.50	0.097	ug/kg
8082 Congeners	3540C	Soil	PCB149 (2,2',3,4',5',6-Hexachlorobiphenyl)	0.50	0.14	ug/kg
8082 Congeners	3540C	Soil	PCB151 (2,2',3,5,5',6-Hexachlorobiphenyl)	0.50	0.080	ug/kg
8082 Congeners	3540C	Soil	PCB153 (2,2',4,4',5,5'-Hexachlorobiphenyl)	0.50	0.18	ug/kg
8082 Congeners	3540C	Soil	PCB156 (2,3,3',4,4',5-Hexachlorobiphenyl)	0.50	0.077	ug/kg
8082 Congeners	3540C	Soil	PCB157 (2,3,3',4,4',5'-Hexachlorobiphenyl)	0.50	0.14	ug/kg
8082 Congeners	3540C	Soil	PCB158 (2,3,3',4,4',6-Hexachlorobiphenyl)	0.50	0.17	ug/kg
8082 Congeners	3540C	Soil	PCB166 (2,3,4,4',5,6-Hexachlorobiphenyl)	0.50	0.36	ug/kg

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**
8082 Congeners	3540C	Soil	PCB167 (2,3',4,4',5,5'-Hexachlorobiphenyl)	0.50	0.11	ug/kg
8082 Congeners	3540C	Soil	PCB168 (2,3',4,4',5',6-Hexachlorobiphenyl)	0.50	0.14	ug/kg
8082 Congeners	3540C	Soil	PCB169 (3,3',4,4',5,5'-Hexachlorobiphenyl)	0.50	0.15	ug/kg
8082 Congeners	3540C	Soil	PCB170 (2,2',3,3',4,4',5-Heptachlorobiphenyl)	0.50	0.16	ug/kg
8082 Congeners	3540C	Soil	PCB174 (2,2',3,3',4,5,6'-Heptachlorobiphenyl)	0.50	0.11	ug/kg
8082 Congeners	3540C	Soil	PCB177 (2,2',3,3',4',5,6-Heptachlorobiphenyl)	0.50	0.13	ug/kg
8082 Congeners	3540C	Soil	PCB18 (2,2',5-Trichlorobiphenyl)	0.50	0.24	ug/kg
8082 Congeners	3540C	Soil	PCB180 (2,2',3,4,4',5,5'-Heptachlorobiphenyl)	0.50	0.076	ug/kg
8082 Congeners	3540C	Soil	PCB183 (2,2',3,4,4',5',6-Heptachlorobiphenyl)	0.50	0.078	ug/kg
8082 Congeners	3540C	Soil	PCB184 (2,2',3,4,4',6,6'-Heptachlorobiphenyl)	0.50	0.066	ug/kg
8082 Congeners	3540C	Soil	PCB187 (2,2',3,4',5,5',6-Heptachlorobiphenyl)	0.50	0.27	ug/kg
8082 Congeners	3540C	Soil	PCB189 (2,3,3',4,4',5,5'-Heptachlorobiphenyl)	0.50	0.11	ug/kg
8082 Congeners	3540C	Soil	PCB194 (2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	0.50	0.076	ug/kg
8082 Congeners	3540C	Soil	PCB195 (2,2',3,3',4,4',5,6-Octachlorobiphenyl)	0.50	0.18	ug/kg
8082 Congeners	3540C	Soil	PCB201 (2,2',3,3',4,5',6,6'-Octachlorobiphenyl)	0.50	0.10	ug/kg
8082 Congeners	3540C	Soil	PCB203 (2,2',3,4,4',5,5',6-Octachlorobiphenyl)	0.50	0.094	ug/kg
8082 Congeners	3540C	Soil	PCB206 (2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	0.50	0.094	ug/kg
8082 Congeners	3540C	Soil	PCB209 (Decachlorobiphenyl)	0.50	0.12	ug/kg
8082 Congeners	3540C	Soil	PCB28 (2,4,4'-Trichlorobiphenyl)	0.50	0.083	ug/kg
8082 Congeners	3540C	Soil	PCB31 (2,4',5-Trichlorobiphenyl)	0.50	0.51	ug/kg
8082 Congeners	3540C	Soil	PCB33 (2',3,4-Trichlorobiphenyl)	0.50	0.18	ug/kg
8082 Congeners	3540C	Soil	PCB37 (3,4,4'-Trichlorobiphenyl)	0.50	0.22	ug/kg
8082 Congeners	3540C	Soil	PCB44 (2,2',3,5'-Tetrachlorobiphenyl)	0.50	0.098	ug/kg
8082 Congeners	3540C	Soil	PCB49 (2,2',4,5'-Tetrachlorobiphenyl)	0.50	0.28	ug/kg
8082 Congeners	3540C	Soil	PCB5 (2,3-Dichlorobiphenyl)	0.50	0.12	ug/kg
8082 Congeners	3540C	Soil	PCB52 (2,2',5,5'-Tetrachlorobiphenyl)	0.50	0.088	ug/kg
8082 Congeners	3540C	Soil	PCB56 (2,3,3',4'-Tetrachlorobiphenyl)	0.50	0.077	ug/kg
8082 Congeners	3540C	Soil	PCB60 (2,3,4,4'-Tetrachlorobiphenyl)	0.50	0.14	ug/kg
8082 Congeners	3540C	Soil	PCB66 (2,3',4,4'-Tetrachlorobiphenyl)	0.50	0.070	ug/kg
8082 Congeners	3540C	Soil	PCB70 (2,3',4',5-Tetrachlorobiphenyl)	0.50	0.11	ug/kg
8082 Congeners	3540C	Soil	PCB74 (2,4,4',5-Tetrachlorobiphenyl)	0.50	0.19	ug/kg
8082 Congeners	3540C	Soil	PCB77 (3,3',4,4'-Tetrachlorobiphenyl)	0.50	0.16	ug/kg
8082 Congeners	3540C	Soil	PCB8 (2,4'-Dichlorobiphenyl)	0.50	0.19	ug/kg
8082 Congeners	3540C	Soil	PCB81 (3,4,4',5-Tetrachlorobiphenyl)	0.50	0.17	ug/kg
8082 Congeners	3540C	Soil	PCB87 (2,2',3,4,5'-Pentachlorobiphenyl)	0.50	0.15	ug/kg

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**
8082 Congeners	3540C	Soil	PCB90 (2,2',3,4',5-Pentachlorobiphenyl)	0.50	0.16	ug/kg
8082 Congeners	3540C	Soil	PCB95 (2,2',3,5',6-Pentachlorobiphenyl)	0.50	0.37	ug/kg
8082 Congeners	3540C	Soil	PCB97 (2,2',3',4,5-Pentachlorobiphenyl)	0.50	0.10	ug/kg
8082 Congeners	3540C	Soil	PCB99 (2,2',4,4',5-Pentachlorobiphenyl)	0.50	0.24	ug/kg
8082 Congeners	3540C	Tissue	PCB1 (2-Chlorobiphenyl)	5.0	5.0	ug/kg
8082 Congeners	3540C	Tissue	PCB101 (2,2',4,5,5'-Pentachlorobiphenyl)	0.50	0.210	ug/kg
8082 Congeners	3540C	Tissue	PCB105 (2,3,3',4,4'-Pentachlorobiphenyl)	0.50	0.075	ug/kg
8082 Congeners	3540C	Tissue	PCB110 (2,3,3',4',6-Pentachlorobiphenyl)	0.50	0.100	ug/kg
8082 Congeners	3540C	Tissue	PCB114 (2,3,4,4',5-Pentachlorobiphenyl)	0.50	0.110	ug/kg
8082 Congeners	3540C	Tissue	PCB118 (2,3',4,4',5-Pentachlorobiphenyl)	0.50	0.089	ug/kg
8082 Congeners	3540C	Tissue	PCB119 (2,3',4,4',6-Pentachlorobiphenyl)	0.50	0.200	ug/kg
8082 Congeners	3540C	Tissue	PCB123 (2',3,4,4',5-Pentachlorobiphenyl)	0.50	0.090	ug/kg
8082 Congeners	3540C	Tissue	PCB126 (3,3',4,4',5-Pentachlorobiphenyl)	0.50	0.120	ug/kg
8082 Congeners	3540C	Tissue	PCB128 (2,2',3,3',4,4'-Hexachlorobiphenyl)	0.50	0.140	ug/kg
8082 Congeners	3540C	Tissue	PCB132 (2,2',3,3',4,6'-Hexachlorobiphenyl)	0.50	0.091	ug/kg
8082 Congeners	3540C	Tissue	PCB138 (2,2',3,4,4',5'-Hexachlorobiphenyl)	0.50	0.110	ug/kg
8082 Congeners	3540C	Tissue	PCB141 (2,2',3,4,5,5'-Hexachlorobiphenyl)	0.50	0.077	ug/kg
8082 Congeners	3540C	Tissue	PCB149 (2,2',3,4',5',6-Hexachlorobiphenyl)	0.50	0.081	ug/kg
8082 Congeners	3540C	Tissue	PCB151 (2,2',3,5,5',6-Hexachlorobiphenyl)	0.50	0.100	ug/kg
8082 Congeners	3540C	Tissue	PCB153 (2,2',4,4',5,5'-Hexachlorobiphenyl)	0.50	0.100	ug/kg
8082 Congeners	3540C	Tissue	PCB156 (2,3,3',4,4',5-Hexachlorobiphenyl)	0.50	0.078	ug/kg
8082 Congeners	3540C	Tissue	PCB157 (2,3,3',4,4',5'-Hexachlorobiphenyl)	0.50	0.160	ug/kg
8082 Congeners	3540C	Tissue	PCB158 (2,3,3',4,4',6-Hexachlorobiphenyl)	0.50	0.074	ug/kg
8082 Congeners	3540C	Tissue	PCB166 (2,3,4,4',5,6-Hexachlorobiphenyl)	0.50	0.066	ug/kg
8082 Congeners	3540C	Tissue	PCB167 (2,3',4,4',5,5'-Hexachlorobiphenyl)	0.50	0.410	ug/kg
8082 Congeners	3540C	Tissue	PCB168 (2,3',4,4',5',6-Hexachlorobiphenyl)	0.50	0.070	ug/kg
8082 Congeners	3540C	Tissue	PCB169 (3,3',4,4',5,5'-Hexachlorobiphenyl)	0.50	0.170	ug/kg
8082 Congeners	3540C	Tissue	PCB170 (2,2',3,3',4,4',5-Heptachlorobiphenyl)	1.0	0.67	ug/kg
8082 Congeners	3540C	Tissue	PCB174 (2,2',3,3',4,5,6'-Heptachlorobiphenyl)	0.50	0.290	ug/kg
8082 Congeners	3540C	Tissue	PCB177 (2,2',3,3',4',5,6-Heptachlorobiphenyl)	0.50	0.170	ug/kg
8082 Congeners	3540C	Tissue	PCB18 (2,2',5-Trichlorobiphenyl)	0.50	0.22	ug/kg
8082 Congeners	3540C	Tissue	PCB180 (2,2',3,4,4',5,5'-Heptachlorobiphenyl)	1.0	0.94	ug/kg
8082 Congeners	3540C	Tissue	PCB183 (2,2',3,4,4',5',6-Heptachlorobiphenyl)	0.50	0.066	ug/kg
8082 Congeners	3540C	Tissue	PCB184 (2,2',3,4,4',6,6'-Heptachlorobiphenyl)	0.50	0.091	ug/kg
8082 Congeners	3540C	Tissue	PCB187 (2,2',3,4',5,5',6-Heptachlorobiphenyl)	0.50	0.450	ug/kg

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**
8082 Congeners	3540C	Tissue	PCB189 (2,3,3',4,4',5,5'-Heptachlorobiphenyl)	0.50	0.110	ug/kg
8082 Congeners	3540C	Tissue	PCB194 (2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	0.50	0.130	ug/kg
8082 Congeners	3540C	Tissue	PCB195 (2,2',3,3',4,4',5,6-Octachlorobiphenyl)	0.50	0.100	ug/kg
8082 Congeners	3540C	Tissue	PCB201 (2,2',3,3',4,5',6,6'-Octachlorobiphenyl)	0.50	0.410	ug/kg
8082 Congeners	3540C	Tissue	PCB203 (2,2',3,4,4',5,5',6-Octachlorobiphenyl)	0.50	0.110	ug/kg
8082 Congeners	3540C	Tissue	PCB206 (2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	0.50	0.098	ug/kg
8082 Congeners	3540C	Tissue	PCB209 (Decachlorobiphenyl)	0.50	0.140	ug/kg
8082 Congeners	3540C	Tissue	PCB28 (2,4,4'-Trichlorobiphenyl)	0.50	0.130	ug/kg
8082 Congeners	3540C	Tissue	PCB31 (2,4',5-Trichlorobiphenyl)	0.50	0.350	ug/kg
8082 Congeners	3540C	Tissue	PCB33 (2',3,4-Trichlorobiphenyl)	0.50	0.170	ug/kg
8082 Congeners	3540C	Tissue	PCB37 (3,4,4'-Trichlorobiphenyl)	0.50	0.200	ug/kg
8082 Congeners	3540C	Tissue	PCB44 (2,2',3,5'-Tetrachlorobiphenyl)	0.50	0.140	ug/kg
8082 Congeners	3540C	Tissue	PCB49 (2,2',4,5'-Tetrachlorobiphenyl)	0.50	0.130	ug/kg
8082 Congeners	3540C	Tissue	PCB5 (2,3-Dichlorobiphenyl)	0.50	0.120	ug/kg
8082 Congeners	3540C	Tissue	PCB52 (2,2',5,5'-Tetrachlorobiphenyl)	0.50	0.110	ug/kg
8082 Congeners	3540C	Tissue	PCB56 (2,3,3',4'-Tetrachlorobiphenyl)	0.50	0.140	ug/kg
8082 Congeners	3540C	Tissue	PCB60 (2,3,4,4'-Tetrachlorobiphenyl)	0.50	0.076	ug/kg
8082 Congeners	3540C	Tissue	PCB66 (2,3',4,4'-Tetrachlorobiphenyl)	0.50	0.100	ug/kg
8082 Congeners	3540C	Tissue	PCB70 (2,3',4',5-Tetrachlorobiphenyl)	0.50	0.150	ug/kg
8082 Congeners	3540C	Tissue	PCB74 (2,4,4',5-Tetrachlorobiphenyl)	0.50	0.130	ug/kg
8082 Congeners	3540C	Tissue	PCB77 (3,3',4,4'-Tetrachlorobiphenyl)	0.50	0.085	ug/kg
8082 Congeners	3540C	Tissue	PCB8 (2,4'-Dichlorobiphenyl)	0.50	0.130	ug/kg
8082 Congeners	3540C	Tissue	PCB81 (3,4,4',5-Tetrachlorobiphenyl)	0.50	0.093	ug/kg
8082 Congeners	3540C	Tissue	PCB87 (2,2',3,4,5'-Pentachlorobiphenyl)	0.50	0.140	ug/kg
8082 Congeners	3540C	Tissue	PCB90 (2,2',3,4',5-Pentachlorobiphenyl)	0.50	0.090	ug/kg
8082 Congeners	3540C	Tissue	PCB95 (2,2',3,5',6-Pentachlorobiphenyl)	0.50	0.400	ug/kg
8082 Congeners	3540C	Tissue	PCB97 (2,2',3',4,5-Pentachlorobiphenyl)	0.50	0.140	ug/kg
8082 Congeners	3540C	Tissue	PCB99 (2,2',4,4',5-Pentachlorobiphenyl)	0.50	0.130	ug/kg
8270C-SIM PAH	3541	Soil	1-Methylnaphthalene	5	0.31	ug/kg
8270C-SIM PAH	3541	Soil	1-Methylphenanthrene	5	0.28	ug/kg
8270C-SIM PAH	3541	Soil	2,3,5-Trimethylnaphthalene	5	0.21	ug/kg
8270C-SIM PAH	3541	Soil	2,6-Dimethylnaphthalene	5	0.36	ug/kg
8270C-SIM PAH	3541	Soil	2-Methylnaphthalene	5	0.39	ug/kg
8270C-SIM PAH	3541	Soil	Acenaphthene	5	0.23	ug/kg
8270C-SIM PAH	3541	Soil	Acenaphthylene	5	0.24	ug/kg

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**
8270C-SIM PAH	3541	Soil	Anthracene	5	0.47	ug/kg
8270C-SIM PAH	3541	Soil	Benz(a)anthracene	5	0.48	ug/kg
8270C-SIM PAH	3541	Soil	Benzo(a)pyrene	5	0.14	ug/kg
8270C-SIM PAH	3541	Soil	Benzo(b)fluoranthene	5	0.25	ug/kg
8270C-SIM PAH	3541	Soil	Benzo(e)pyrene	5	0.18	ug/kg
8270C-SIM PAH	3541	Soil	Benzo(g,h,i)perylene	5	0.64	ug/kg
8270C-SIM PAH	3541	Soil	Benzo(k)fluoranthene	5	0.15	ug/kg
8270C-SIM PAH	3541	Soil	Biphenyl	5	0.40	ug/kg
8270C-SIM PAH	3541	Soil	Carbazole	5	2.3	ug/kg
8270C-SIM PAH	3541	Soil	Chrysene	5	0.25	ug/kg
8270C-SIM PAH	3541	Soil	Dibenz(a,h)anthracene	5	0.28	ug/kg
8270C-SIM PAH	3541	Soil	Dibenzofuran	5	0.59	ug/kg
8270C-SIM PAH	3541	Soil	Dibenzothiophene	5	0.21	ug/kg
8270C-SIM PAH	3541	Soil	Fluoranthene	5	0.61	ug/kg
8270C-SIM PAH	3541	Soil	Fluorene	5	0.50	ug/kg
8270C-SIM PAH	3541	Soil	Indeno(1,2,3-cd)pyrene	5	0.16	ug/kg
8270C-SIM PAH	3541	Soil	Naphthalene	5	0.37	ug/kg
8270C-SIM PAH	3541	Soil	Pentachlorophenol	200	2.9	ug/kg
8270C-SIM PAH	3541	Soil	Perylene	5	0.32	ug/kg
8270C-SIM PAH	3541	Soil	Phenanthrene	5	0.75	ug/kg
8270C-SIM PAH	3541	Soil	Pyrene	5	0.37	ug/kg
8270C-SIM PAH	3541	Tissue	1-Methylnaphthalene	0.5	0.30	ug/kg
8270C-SIM PAH	3541	Tissue	1-Methylphenanthrene	0.5	0.082	ug/kg
8270C-SIM PAH	3541	Tissue	2,3,5-Trimethylnaphthalene	0.5	0.24	ug/kg
8270C-SIM PAH	3541	Tissue	2,6-Dimethylnaphthalene	0.5	0.41	ug/kg
8270C-SIM PAH	3541	Tissue	2-Methylnaphthalene	1.0	0.44	ug/kg
8270C-SIM PAH	3541	Tissue	Acenaphthene	0.5	0.11	ug/kg
8270C-SIM PAH	3541	Tissue	Acenaphthylene	0.5	0.069	ug/kg
8270C-SIM PAH	3541	Tissue	Anthracene	0.5	0.065	ug/kg
8270C-SIM PAH	3541	Tissue	Benz(a)anthracene	0.5	0.066	ug/kg
8270C-SIM PAH	3541	Tissue	Benzo(a)pyrene	0.5	0.081	ug/kg
8270C-SIM PAH	3541	Tissue	Benzo(b)fluoranthene	0.5	0.070	ug/kg
8270C-SIM PAH	3541	Tissue	Benzo(e)pyrene	0.5	0.062	ug/kg
8270C-SIM PAH	3541	Tissue	Benzo(g,h,i)perylene	0.5	0.073	ug/kg
8270C-SIM PAH	3541	Tissue	Benzo(k)fluoranthene	0.5	0.056	ug/kg

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**
8270C-SIM PAH	3541	Tissue	Biphenyl	0.5	0.17	ug/kg
8270C-SIM PAH	3541	Tissue	Carbazole	0.5	0.13	ug/kg
8270C-SIM PAH	3541	Tissue	Chrysene	0.5	0.076	ug/kg
8270C-SIM PAH	3541	Tissue	Dibenz(a,h)anthracene	0.5	0.059	ug/kg
8270C-SIM PAH	3541	Tissue	Dibenzofuran	0.5	0.13	ug/kg
8270C-SIM PAH	3541	Tissue	Dibenzothiophene	0.5	0.074	ug/kg
8270C-SIM PAH	3541	Tissue	Fluoranthene	0.5	0.090	ug/kg
8270C-SIM PAH	3541	Tissue	Fluorene	0.5	0.15	ug/kg
8270C-SIM PAH	3541	Tissue	Indeno(1,2,3-cd)pyrene	0.5	0.064	ug/kg
8270C-SIM PAH	3541	Tissue	Naphthalene	1.0	0.40	ug/kg
8270C-SIM PAH	3541	Tissue	Perylene	0.5	0.064	ug/kg
8270C-SIM PAH	3541	Tissue	Phenanthrene	0.5	0.36	ug/kg
8270C-SIM PAH	3541	Tissue	Pyrene	0.5	0.098	ug/kg
8270C-SIM PAH	3520C	Water	1-Methylnaphthalene	0.02	0.0035	ug/L
8270C-SIM PAH	3520C	Water	1-Methylphenanthrene	0.02	0.0041	ug/L
8270C-SIM PAH	3520C	Water	2,3,5-Trimethylnaphthalene	0.02	0.0050	ug/L
8270C-SIM PAH	3520C	Water	2,6-Dimethylnaphthalene	0.02	0.0022	ug/L
8270C-SIM PAH	3520C	Water	2-Methylnaphthalene	0.02	0.0023	ug/L
8270C-SIM PAH	3520C	Water	Acenaphthene	0.02	0.0044	ug/L
8270C-SIM PAH	3520C	Water	Acenaphthylene	0.02	0.0034	ug/L
8270C-SIM PAH	3520C	Water	Anthracene	0.02	0.0036	ug/L
8270C-SIM PAH	3520C	Water	Benz(a)anthracene	0.02	0.0026	ug/L
8270C-SIM PAH	3520C	Water	Benzo(a)pyrene	0.02	0.0043	ug/L
8270C-SIM PAH	3520C	Water	Benzo(b)fluoranthene	0.02	0.0023	ug/L
8270C-SIM PAH	3520C	Water	Benzo(e)pyrene	0.02	0.0040	ug/L
8270C-SIM PAH	3520C	Water	Benzo(g,h,i)perylene	0.02	0.0029	ug/L
8270C-SIM PAH	3520C	Water	Benzo(k)fluoranthene	0.02	0.0025	ug/L
8270C-SIM PAH	3520C	Water	Biphenyl	0.02	0.0024	ug/L
8270C-SIM PAH	3520C	Water	Carbazole	0.02	0.0045	ug/L
8270C-SIM PAH	3520C	Water	Chrysene	0.02	0.0034	ug/L
8270C-SIM PAH	3520C	Water	Dibenz(a,h)anthracene	0.02	0.0025	ug/L
8270C-SIM PAH	3520C	Water	Dibenzofuran	0.02	0.0046	ug/L
8270C-SIM PAH	3520C	Water	Dibenzothiophene	0.02	0.0038	ug/L
8270C-SIM PAH	3520C	Water	Fluoranthene	0.02	0.0044	ug/L
8270C-SIM PAH	3520C	Water	Fluorene	0.02	0.0038	ug/L

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**
8270C-SIM PAH	3520C	Water	Indeno(1,2,3-cd)pyrene	0.02	0.0026	ug/L
8270C-SIM PAH	3520C	Water	Naphthalene	0.02	0.0030	ug/L
8270C-SIM PAH	3520C	Water	Pentachlorophenol	1	0.017	ug/L
8270C-SIM PAH	3520C	Water	Perylene	0.02	0.0050	ug/L
8270C-SIM PAH	3520C	Water	Phenanthrene	0.02	0.0050	ug/L
8270C-SIM PAH	3520C	Water	Pyrene	0.02	0.0035	ug/L
8330B	Method	Tissue	1,3,5-Trinitrobenzene	2.0	0.0044	mg/kg
8330B	Method	Tissue	1,3-Dinitrobenzene	2.0	0.0044	mg/kg
8330B	Method	Tissue	2,4,6-Trinitrotoluene	2.0	0.0066	mg/kg
8330B	Method	Tissue	2,4-Dinitrotoluene	2.0	0.0057	mg/kg
8330B	Method	Tissue	2,6-Dinitrotoluene	2.0	0.0086	mg/kg
8330B	Method	Tissue	2-Amino-4,6-dinitrotoluene	2.0	0.006	mg/kg
8330B	Method	Tissue	2-Nitrotoluene	2.0	0.018	mg/kg
8330B	Method	Tissue	3,5-Dinitroaniline	2.0	0.0047	mg/kg
8330B	Method	Tissue	3-Nitrotoluene	2.0	0.011	mg/kg
8330B	Method	Tissue	4-Amino-2,6-dinitrotoluene	2.0	0.012	mg/kg
8330B	Method	Tissue	4-Nitrotoluene	2.0	0.013	mg/kg
8330B	Method	Tissue	HMX	2.0	0.0073	mg/kg
8330B	Method	Tissue	Nitrobenzene	2.0	0.0069	mg/kg
8330B	Method	Tissue	RDX	2.0	0.0059	mg/kg
8330B	Method	Tissue	TETRYL	2.0	0.012	mg/kg
Organotins	Method	Sediment	Di-n-butyltin	1	0.24	ug/kg
Organotins	Method	Sediment	n-Butyltin	1	0.21	ug/kg
Organotins	Method	Sediment	Tetra-n-butyltin	1	0.47	ug/kg
Organotins	Method	Sediment	Tri-n-butyltin	1	0.3	ug/kg
Organotins	Method	Water	Di-n-butyltin	0.05	0.0073	ug/L
Organotins	Method	Water	n-Butyltin	0.05	0.029	ug/L
Organotins	Method	Water	Tetra-n-butyltin	0.05	0.038	ug/L
Organotins	Method	Water	Tri-n-butyltin	0.05	0.012	ug/L
Organotins	Method	Tissue	Di-n-butyltin	1	0.091	ug/kg
Organotins	Method	Tissue	n-Butyltin	1	0.20	ug/kg
Organotins	Method	Tissue	Tetra-n-butyltin	1	0.18	ug/kg
Organotins	Method	Tissue	Tri-n-butyltin	1	0.33	ug/kg

a Method Detection Limits are subject to change as new MDL studies are completed.

b For tissue, units are dry weight basis for metals and wet weight basis for organics.

SELECTED MARINE WATER, SEDIMENT, AND TISSUE ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units[b]**

TCLP ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
6010B	3010A	Water	Arsenic	0.1	0.02	mg/L
6010B	3010A	Water	Barium	1	0.3	mg/L
6010B	3010A	Water	Cadmium	0.01	0.002	mg/L
6010B	3010A	Water	Chromium	0.01	0.003	mg/L
6010B	3010A	Water	Lead	0.05	0.03	mg/L
7470A	3010A	Water	Mercury	0.001	0.0002	mg/L
6010B	3010A	Water	Selenium	0.1	0.03	mg/L
6010B	3010A	Water	Silver	0.02	0.008	mg/L
8081A	3535	Water	Chlordane	0.005		mg/L
8081A	3535	Water	Endrin	0.0005		mg/L
8081A	3535	Water	gamma-BHC (Lindane)	0.0005		mg/L
8081A	3535	Water	Heptachlor	0.0005		mg/L
8081A	3535	Water	Methoxychlor	0.001		mg/L
8081A	3535	Water	Toxaphene	0.01		mg/L
8151A	Method	Water	2,4,5-TP (Silvex)	20		ug/L
8151A	Method	Water	2,4-D	100		ug/L
8260B	5030B	Water	1,1-Dichloroethene	0.2	0.119	mg/L
8260B	5030B	Water	1,2-Dichloroethane (EDC)	0.2	0.114	mg/L
8260B	5030B	Water	1,4-Dichlorobenzene	0.2	.0869	mg/L
8260B	5030B	Water	2-Butanone (MEK)	8.0	3.22	mg/L
8260B	5030B	Water	Benzene	0.2	0.105	mg/L
8260B	5030B	Water	Carbon Tetrachloride	0.2	0.123	mg/L
8260B	5030B	Water	Chlorobenzene	0.2	0.0933	mg/L
8260B	5030B	Water	Chloroform	0.2	0.0958	mg/L
8260B	5030B	Water	Tetrachloroethene (PCE)	0.2	0.109	mg/L
8260B	5030B	Water	Trichloroethene (TCE)	0.2	0.118	mg/L
8260B	5030B	Water	Vinyl Chloride	0.08	0.0211	mg/L
8270C	3510C	Water	2,4,5-Trichlorophenol	0.1	0.00869	mg/L
8270C	3510C	Water	2,4,6-Trichlorophenol	0.1	0.00687	mg/L
8270C	3510C	Water	2,4-Dinitrotoluene	0.1	0.0130	mg/L
8270C	3510C	Water	2-Methylphenol	0.1	0.00854	mg/L
8270C	3510C	Water	3- and 4-Methylphenol Coelution	0.1	0.00468	mg/L
8270C	3510C	Water	Hexachlorobenzene	0.1	0.00936	mg/L
8270C	3510C	Water	Hexachlorobutadiene	0.1	0.00633	mg/L
8270C	3510C	Water	Hexachloroethane	0.1	0.00471	mg/L

TCLP ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
8270C	3510C	Water	Nitrobenzene	0.1	0.00788	mg/L
8270C	3510C	Water	Pentachlorophenol	0.25	0.0107	mg/L
8270C	3510C	Water	Pyridine	0.5	0.249	mg/L

a Method Detection Limits are subject to change as new MDL studies are completed.

MICROBIOLOGICAL ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**Method Reporting Limit**	**Method Detection Limit[a]**	**Units**
SM 9215B			Heterotrophic Plate Count (Pour)	1	NA	CFU/mL
SM 9215C			Heterotrophic Plate Count (Spread)	1	NA	CFU/mL
SM 9221B			Coliform, Total (MPN)	2	NA	MPN/100mL
SM 9221E			Coliform, Fecal (MPN)	2	NA	MPN/100mL
SM 9223B			Coliform (Colilert)	Presence/100mL	NA	NA
SM 9230B			Enterococcus	2	NA	MPN/100mL
SM 9230B			Fecal Streptococcus	2	NA	MPN/100mL
SM 10200H			Chlorophyll A	0.8	0.3	mg/L

a Method Detection Limits are subject to change as new MDL studies are completed.

GENERAL CHEMISTRY/WATER CHEMISTRY ANALYSES						
Method	**Prep Method**	**Matrix**	Analyte	**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
1650C	NA	Water	Absorbable Organic Halides	78-116	78-116	20
305.1/*SM* 2310B 4a	NA	Water	Acidity as $CaCO_3$	85-115	NA	20
310.1 / *SM* 2320B	NA	Water	Alkalinity as $CaCO_3$	85-115	NA	20
350.1M	SOP	Soil	Ammonia as Nitrogen	58-131	66-127	32
350.1	NA	Water	Ammonia as Nitrogen	90-110	90-110	20
350.3M	SOP	Soil	Ammonia as Nitrogen	85-115	75-125	20
350.3/*SM* 4500NH3 G	NA	Water	Ammonia as Nitrogen	73-117	84-110	20
405.1/*SM* 5210B	NA	Water	Biological Oxygen Demand	85-115	NA	20
300.1	NA	Water	Bromate	85-115	75-125	20
300.0M	SOP	Soil	Bromide	90-110	80-120	20
300.0	NA	Water	Bromide	90-110	80-120	20
300.1	NA	Water	Bromide	85-115	75-125	20
410.1 and 410.2M	SOP	Soil	Chemical Oxygen Demand	85-115	75-125	20
410.1/410.2/*SM* 5220C	NA	Water	Chemical Oxygen Demand	85-115	75-125	20
300.0M	SOP	Soil	Chloride	90-110	80-120	20
300.0	NA	Water	Chloride	90-110	80-120	20
325.3/*SM* 4500Cl- C	NA	Water	Chloride, Titrimetric	85-115	75-125	20
9252	NA	Water	Chloride, Titrimetric	85-115	75-125	20
330.4/*SM* 4500 Cl- F	NA	Water	Chlorine, Total Residual	85-115	NA	20
300.1	NA	Water	Chlorite	85-115	75-125	20
110.2/*SM* 2120B	NA	Water	Color	85-115	NA	20
120.1 / *SM* 2510B	NA	Water	Conductivity	85-115	NA	20
1110	NA	Liquid	Corrosivity	NA	NA	20
9040	NA	Water	Corrosivity (pH)	NA	NA	20
335.2 / 335.4	SOP	Soil	Cyanide, Total	85-115	75-125	20
335.4	NA	Water	Cyanide, Total	90-110	90-110	20
9010B/9012A		Soil	Cyanide, Total and Amenable	85-115	75-125	20
9010B/9012A		Water	Cyanide, Total and Amenable	85-115	75-125	20
335.1M	SOP	Soil	Cyanides Amenable to Chlorination	85-115	75-125	20
335.1/*SM* 4500CN G	NA	Water	Cyanides Amenable to Chlorination	85-115	75-125	20
300.1	NA	Water	Dichloroacetate (surrogate) 90-115	NA	NA	NA
1020	NA	Soil	Flashpoint, Setaflash	NA	NA	20
1020	NA	Water	Flashpoint, Setaflash	NA	NA	20
300.0M	SOP	Soil	Fluoride	90-110	80-120	20
300.0	NA	Water	Fluoride	90-110	80-120	20
340.2M	SOP	Soil	Fluoride	85-115	75-125	20
340.2 / *SM* 4500-F⁻C	NA	Water	Fluoride	85-115	75-125	20

GENERAL CHEMISTRY/WATER CHEMISTRY ANALYSES						
Method	**Prep Method**	**Matrix**	Analyte	**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
340.1M	SOP	Soil	Fluoride, Bellack Distillation	85-115	75-125	20
340.1	NA	Water	Fluoride, Bellack Distillation	85-115	75-125	20
130.2 / *SM* 2340C	NA	Water	Hardness as $CaCO_3$	85-115	75-125	20
7196A	3060A	Soil	Hexavalent Chromium	80-120	75-125	20
7196A	Method	Water	Hexavalent Chromium	85-115	85-115	20
300.0M	SOP	Soil	Nitrate as Nitrogen	90-110	80-120	20
300.0	NA	Water	Nitrate as Nitrogen	90-110	80-120	20
300.0M	SOP	Soil	Nitrite as Nitrogen	90-110	80-120	20
300.0	NA	Water	Nitrite as Nitrogen	90-110	80-120	20
354.1/*SM* 4500NO2 B	NA	Water	Nitrite as Nitrogen, Colorimetric	85-115	75-125	20
353.2M	SOP	Soil	Nitrogen, Nitrate + Nitrite as Nitrogen	85-115	75-125	20
353.2	NA	Water	Nitrogen, Nitrate + Nitrite as Nitrogen	90-110	90-110	20
351.4M	SOP	Soil	Nitrogen, Total Kjeldahl	70-108	38-138	20
351.4/ASTM D3590-89	NA	Water	Nitrogen, Total Kjeldahl	85-115	75-125	20
365.3M	SOP	Soil	Orthophosphate as Phosphorus	85-115	75-125	20
365.3	NA	Water	Orthophosphate as Phosphorus	85-115	75-125	20
SM 4500P-F	NA	Water	Orthophosphate as Phosphorus	85-115	75-125	20
ASTM D1498	NA	Water	Oxidation-Reduction Potential	NA	NA	20
9095	NA	Soil	Paint Filter Test	85-115	NA	NA
314.0	NA	Water	Perchlorate	85-115	80-120	20
150.1/*SM* 4500H+ B	NA	Water	pH	85-115	NA	NA
9045C	NA	Soil	pH	NA	NA	20
420.1M	SOP	Soil	Phenolics, Total	85-115	75-125	20
420.1	NA	Water	Phenolics, Total	89-110	74-121	20
365.3M	SOP	Soil	Phosphorus, Total	85-115	75-125	20
365.3	NA	Water	Phosphorus, Total	85-115	75-125	20
160.5/*SM* 2540F	NA	Water	Solids, Settleable	NA	NA	20
160.3M	NA	Soil	Solids, Total	NA	NA	20
160.3/*SM* 2540B	NA	Water	Solids, Total	88-112	NA	10
160.1 / *SM* 2540C	NA	Water	Solids, Total Dissolved (Filterable)	85-115	NA	10
160.2/*SM* 2540D	NA	Water	Solids, Total Suspended (Nonfilterable)	85-115	NA	10
160.4	NA	Soil	Solids, Volatile	85-115	NA	20
160.4	NA	Water	Solids, Volatile	85-115	NA	20
300.0M	SOP	Soil	Sulfate	90-110	80-120	20
300.0	NA	Water	Sulfate	90-110	80-120	20
376.1/*SM* 4500S2- F	NA	Water	Sulfide	85-115	75-125	20
376.2/*SM* 4500S2- D	NA	Water	Sulfide	85-115	75-125	20
9030M	Method	Soil	Sulfides	51-125	46-144	43

GENERAL CHEMISTRY/WATER CHEMISTRY ANALYSES						
Method	**Prep Method**	**Matrix**	Analyte	**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
PSEP	PSEP	Soil	Sulfides	51-125	46-144	43
821/R-91-100 Draft	Method	Sediment	Sulfides, Acid Volatile	62-109	66-117	45
9030	NA	Water	Sulfides	60-130	60-130	20
377.1/*SM* 4500SO32-B	NA	Water	Sulfite	NA	NA	20
425.1/*SM* 5540C	NA	Water	Surfactants (MBAS)	85-115	60-130	20
SM 5550B	NA	Water	Tannin and Lignin	85-115	75-125	20
ASTM D4129-82M	NA	Soil	Total Organic Carbon	74-123	75-114	27
415.1/*SM* 5310C	NA	Water	Total Organic Carbon	69-136	49-156	33
9060A	NA	Water	Total Organic Carbon	63-141	77-112	36
9020	NA	Water	Total Organic Halides	87-113	82-121	20
180.1	NA	Water	Turbidity	85-115	NA	20

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Quality Manual

October 14, 2008
(Twenty-ninth Revision)

This Quality Manual has been read, verified, and is approved by the Management of Summit Environmental Technologies, Inc. with the laboratory's approved signatories.

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President
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10/14/08
Date

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Table of Contents

CHAPTER	Page
PREFACE	3
QUALITY POLICY STATEMENT	4
CHAPTER 1- Organization	5
CHAPTER 2- Staff	7
CHAPTER 3- S.E.T. Organizational Chart	10
CHAPTER 4- Facilities and Equipment	11
CHAPTER 5-Laboratory Instrumentation and Equipment	13
CHAPTER 6- Document Control	18
CHAPTER 7- Sample Handling and Chain of Custody	19
CHAPTER 8- Analytical Methods	26
CHAPTER 9- Quality System	34
CHAPTER 10- Data Reduction Validation and Reporting	40
CHAPTER 11- Internal Performance and System Audit	49
CHAPTER 12- Employee Training	55
CHAPTER 13- Quality System Review by Management	57
CHAPTER 14- Lab Safety	58
APPENDIX	59
QC-1 Instrument Preventative Maintenance Determination	60
QC-2 Method Detection Limit Determination	63
QC-3 Quality Control Limits Calculations	64
QC-4 Internal Quality Control Checks	67
QC-5 Reporting Limits	71

Preface

The Summit Environmental Technologies, Inc., heretofore known as the laboratory, Quality Manual is a collection of guidelines provided for clients as well as for federal, state, local and private agencies.

The Quality Manual outlines the basic methods and procedures in performing laboratory analytical services. Laboratory analyses are performed by competent technical personnel who follow well-established techniques, using modern and reliable analytical instruments. Standard Operating Procedures (SOPs) give more concise details concerning analytical methods and laboratory administrative procedures.

Summit Environmental Technologies, Inc. utilizes the technical and administrative procedures contained in the following documents:

Code of Federal Regulations Title 40

"Methods of Chemical Analysis of Water and Wastes", EPA-60014-79-020.

"Standard Methods for Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, 18th - 21st Editions.

"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" USEPA, SW-846, Updates III, IVA, IVB USEPA

"Test Methods for Chemical Analysis of Municipal and Industrial Wastewater", EPA 600/4-82-057, July 1982.

"American Society of Testing and Materials (ASTM)"

"A2LA Environmental Program Requirements", February 2007.

"National Environmental Laboratory Accreditation Conference", July 2003.

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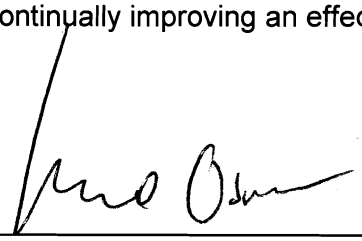
Summit Environmental Technologies, Inc.
Quality Policy Statement

Summit Environmental Technologies, Inc. strives to produce analytical data that is of the highest standards in quality and accuracy. To this end, Summit Environmental Technologies, Inc. management has a commitment to offer to our clients the best in quality service and analysis.

Summit Environmental Technologies, Inc. will perform all analyses in accordance with the method specified by the client and all of the associated Quality Control and Quality Assurance samples necessary to produce an analytical report that is complete, accurate and precise. Summit Environmental Technologies, Inc. will maintain compliance with the requirements set forth in ISO/IEC 17025, and NELAC. Summit Environmental Technologies, Inc. assures that the data reported are within known probability limits of accuracy and precision.

All management and laboratory personnel must read and follow the Quality Manual and relevant Standard Operating Procedures (SOPs) and analytical methods as they pertain to the policies and procedures set forth by ISO/IEC 17025, and NELAC. All analysts are trained and made familiar with all the methods, SOPs, and QA/QC requirements for the analyses performed in their area of operation. Each analyst is responsible to ensure that not only the correct QA/QC is analyzed with the sample, but that all the QA/QC parameters meet laboratory and/or method criteria. No report will be delivered to our clients without passing all of the quality parameters deemed necessary to ensure that the report is accurate and complete.

The laboratory management staff is committed to developing, implementing, and continually improving an effective management system.



Dr. Mo Osman
President
Summit Environmental Technologies, Inc.

CHAPTER 1

Organization

1.1 IDENTIFICATION AND ORGANIZATION

11.1.1 Summit Environmental Technologies, Inc. is a legally responsible entity, incorporated and governed by the State of Ohio. The laboratory's Federal ID number is 34-1773575. The laboratory's EPA ID number is OH00923. The laboratory is located at 3310 Win Street, Cuyahoga Falls, Ohio, 44223, Summit Environmental Technologies, Inc. and is a privately owned commercial laboratory.

11.1.2 All work performed at the Summit Environmental Technologies, Inc. site in Cuyahoga Falls, Ohio is governed by the requirements issued in ISO/IEC 17025 as adopted by the American Association of Laboratory Accreditation (A2LA) and The National Environmental Laboratory Accreditation Conference (NELAC) Institute (TNI). The laboratory is also governed by the individual state accreditations it maintains.

11.1.3 The ratio of the managerial staff to the technical staff is such that there is adequate supervision to ensure adherence to laboratory analytical methods and procedures.

11.1.4 All managerial and technical personnel have the ability to implement, maintain, and improve the management system and to identify any departures from the normal management system of the laboratory. All managerial and technical personnel have the ability to prevent and minimize any departure from normal laboratory operations.

11.1.5 It is the policy of Summit Environmental Technologies, Inc. not to be willfully involved in any illegal or controversial issue that may discredit or otherwise injure the name of the laboratory. It is the laboratory's intent to provide accurate and compliant information to its clients. If an illegal or non-compliant issue is discovered by the laboratory, it will disclose the issue to all concerned and immediately implement corrective action to correct the problem.

1.2 ACCREDITATIONS

Summit Environmental Technologies, Inc. is an accredited environmental laboratory by the following agencies:

1. The American Association for Laboratory Accreditation (A2LA) (Accreditation Number 0724.01)
2. National Environmental Laboratory Accreditation Program (NELAP) Certification through the State of Florida (I.D. E87688)
3. The State of Ohio Voluntary Action Program (CL0052)
4. The State of Arkansas (88-00735)
5. The State of California (07256CA)
6. The State of Georgia (0724.01)
7. The State of Indiana (C-H-13)
8. The State of Kansas (E-10347)
9. The State of Kentucky (0003)
10. The State of Louisiana (04061)
12. The State of Minnesota (039-999-385)
13. The State of New Jersey (OH006)
14. The State of New York (11777)
15. The State of North Carolina (631)
16. The State of Oklahoma (9940)
17. The State of Pennsylvania (68-1335)
18. The State of South Carolina (92016001)
19. The State of Tennessee (0724.01)
20. The State of Utah (3302538211)
21. The State of Washington (C2014)
22. The State of West Virginia (248)
23. The State of Wisconsin (399013010)
24. The State of Oregon (OH200001-001)

CHAPTER 2

Staff

2.1 PRESIDENT

2.1.1 The president is a full time member of the managerial staff and is responsible for the entire operation of the laboratory. He institutes policy and determines the business plan for the laboratory. He maintains sales and client services and determines marketing strategies for the laboratory. He also oversees the financial aspect of the laboratory. He has the final decision concerning laboratory policy and/or procedures, and he oversees the condition and upkeep of the physical plant. The President has the authority to approve and sign all analytical reports in the Laboratory Manager's absence. The Laboratory Manager assumes the President's duties in his absence.

2.2 LABORATORY MANAGER

2.2.1 The Laboratory Manager is a full time member the managerial staff and reports directly to the President. The Laboratory Manager is responsible for assuring that daily operations of the laboratory are performed within appropriate guidelines. He/she provides technical information required for routine and new analytical procedures to the laboratory staff and clients. He/she checks for accurate transcription before data are reported to clients. The Laboratory Manager interacts with the clients and the regulatory personnel to insure that appropriate procedures are followed. The Laboratory Manager approves and signs all analytical reports. The President assumes the Laboratory Manager's duties in his absence.

2.3 QUALITY MANAGER

2.3.1 The Quality Manager reports directly to the President. The Quality Manager has the responsibility and authority for ensuring that the laboratory quality system is implemented and followed at all times. He/she, defines, develops, and maintains the quality assurance programs. He/she monitors QA activities, conducts audits, investigates non-compliance problems, and takes or recommends actions on improvements. He/she maintains, identifies, and conducts quality training programs for employees, and reports and documents QA/QC problems. He/she monitors QC checks and implements protocol adjustments as necessary. The Laboratory Manager is designated the deputy Quality Manager, and will have the same powers as the Quality Manager in his/her absence.

2.4 CHEMISTS AND TECHNICIANS

2.4.1 Chemists and Technicians perform analysis and data recording in accordance with the Summit Environmental QA program. Senior analysts are responsible for management of section group personnel and ensure that all procedures are conducted in compliance with regulatory guidelines. They also perform data validation and document quality control procedures. Minor preventive maintenance functions are performed by Chemists and Technicians.

2.5 SUPPORT STAFF

2.5.1 Support staff includes report generation staff, login staff and office staff. These individuals perform various ancillary functions with regard to the operation of the laboratory. The login staff accepts logs in and stores samples for the laboratory. They also prepare and send out sample bottle requests and contact clients with questions and problems concerning their sample submittals.

2.5.2 The report staff generates the analytical reports from the LIMS. They are also responsible for faxing results to the client and filing all completed and in process analytical report folders. They are occasionally required to respond to client phone calls and offer assistance in non-technical issues.

2.5.3 The office staff is responsible for purchasing, invoicing, payroll, and other clerical duties necessary for the operation of the laboratory.

2.6 GENERAL STAFF FEATURES

2.6.1 The managerial and technical staffs have the competence and ability to carry out their duties and to identify any non-conformance or departures from normal laboratory procedures. All staff members have the ability to conduct corrective actions to rectify any departures from normal laboratory procedures.

2.6.2 Staff members of Summit Environmental Technologies, Inc. are to avoid the acceptance of gifts and gratuities from outside parties in order to obtain special favors or increased attention to their projects. No employee of the laboratory is to pressure another employee into improper or non-compliant situations in order to gain preferential treatment in the course of normal laboratory business.

2.6.3 Each employee is instructed in the rights of the client with regard to confidential and proprietary information. No employee is to divulge any client information to a third party without the written consent of the client. Consult the laboratory SOP for Client Confidentiality for further details. Employees are also bound by the laboratory's ethics policy to ensure correct and compliant analytical data.

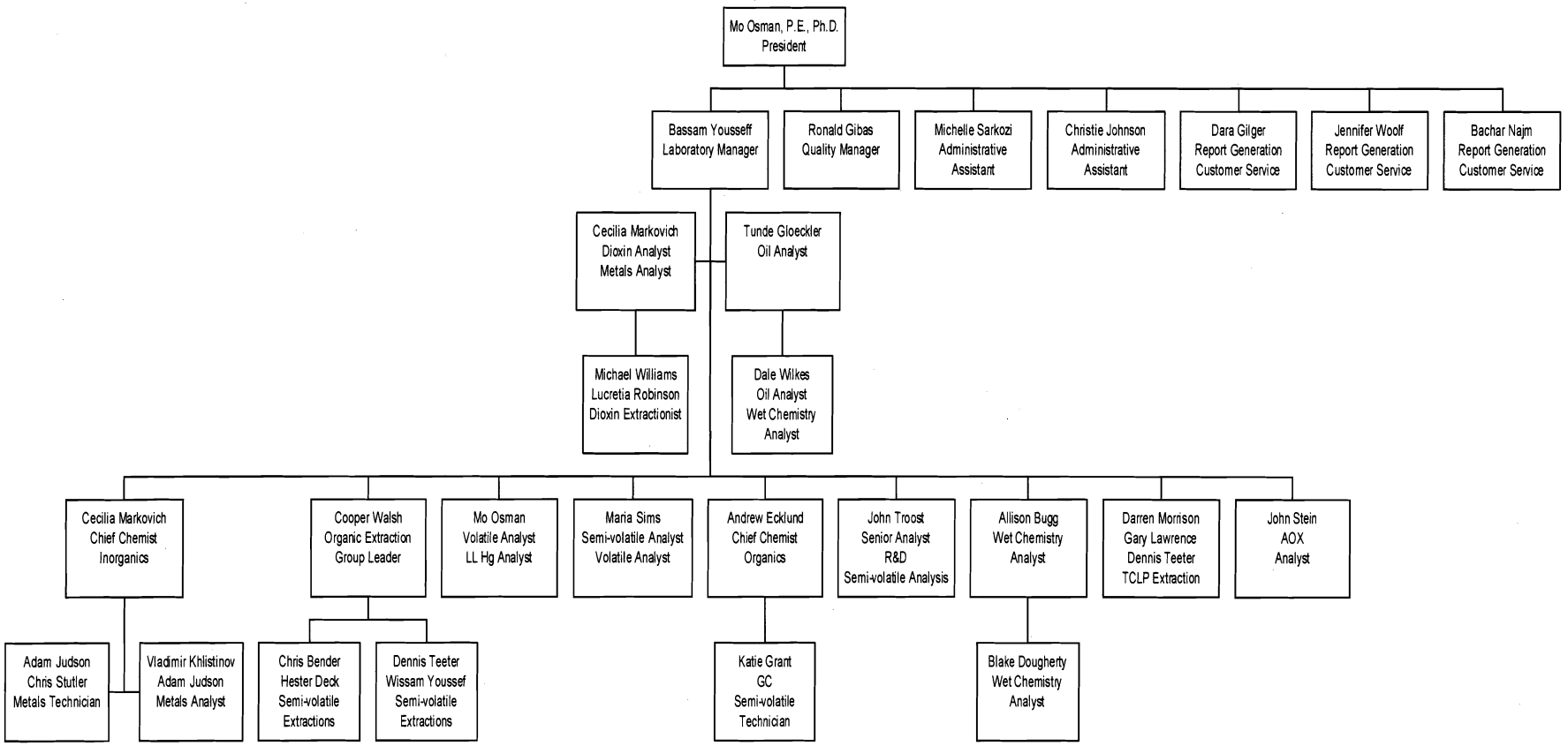
2.6.4 Signatures of all laboratory staff are kept on file in the Quality Manager's office.

2.7 PERSONNEL RECORDS

Records covering the education, training, and technical experience of the laboratory staff are maintained by the Quality Manager.

CHAPTER 3

Summit Environmental Technologies, Inc.
Organizational Chart



CHAPTER 4

Facilities and Equipment

4.1 INSTRUMENT INVENTORY

Instrument	Model	Manufacturer	Quantity
GC/MS w/ purge/trap and AS (LSC2016)	5890/5970	HP	3
GC/MS w/ AS (7673A)	5890/5970	HP	2
GC/MS w/ purge and trap (Velocity XPT)	6890/5973	HP	1
SVOA GC/MS	5890/5970	HP	3
SVOA GC/MS	7890/5975	Agilent	1
Hi Res GC/MS	Micromass	Waters	1
GC/FID/PID	5890 Series II	HP	1
GC/NPD	5890 Series II	HP	1
GC/ECD	5890 Series II	HP	4
GC/FID	5890 Series II	HP	6
GC/PFD	5890 Series II	5890 Series II	1
Closed System Purge and Trap	Archon Tekmar	Dynatech Varian	4
HPLC	Primus	HP	1
HPLC	Summit	Dionex	1
ICP	61E	Thermo Jerrel Ash	1
ICP	Plasma 400	Perkin Elmer	1
ICP Trace	61E	Thermo Jerrel Ash	1
AA Graphite Furnace	Z5100	Perkin Elmer	1
AA w/Flame/Cold Vapor	2380	Perkin Elmer	1
Mercury Analyzer	1600	CETAC	1
Low Level Mercury	Series 2600	Tekran	1
Evaporator	RapidVap N2	Labconco	3
Evaporator	TurboVap II	Caliper Life	2
TOC	TOC-VCPN/TNM-1	Shimadzu	1
Bomb Calorimeter	1341EB	Parrr	1
Solvent Extractor	ASE200	Dionex	1
Sonicator	Virsonic 475	VirTis	1
TPH Analyzer	HC-404	Buck	1
AOX Analyzer	multi X 2000	IDC Analytical System	3
Ion Chromatograph	DX-120	Dionex	1
Ion Chromatograph	ICS-2000	Dionex	1

4.1 INSTRUMENT INVENTORY (CONT)

Bomb Calorimeter	6300	Parr	1
Vacuum Distillation	D1160	B/R Instruments	1
XRF	Spectro 200	Spectro	1
Flash Point	PMA4	Petrotest	1
Flash Point	K16200	Koehler	1
Coulometer	831	Metrohm	1
Viscosity Bath	KV4000	Koehler	1
Distillation Unit	K45000	Koehler	1
Copper Corrosion	K25330	Koehler	1
Oil Comparator	K13200	Koehler	1
Ramsbottom Carbon Residue	K27100	Koehler	1

4.2 LABORATORY FACILITIES AND SAFETY

- The laboratory has adequate ventilation to exhaust fumes produced by analysis and chemicals handled in the laboratory.
- Temperature within the laboratory is maintained within the limits required for proper operation of instruments.
- Workbench space within the laboratory is adequate.
- Fire extinguishers are available.
- Occupational safety and health laws are posted and observed.

4.3 LABORATORY AREAS

Total area of the laboratory is approximately 21,000 square feet. All activities within the laboratory are segregated and divided as follows:

- Administration and customer service area.
- Sample login area.
- Chemical storage area.
- Sample storage area.
- Metals preparation area.
- Metals analysis area.
- Volatiles analysis area.
- Semi-Volatiles extraction area.
- Semi-Volatiles analysis area
- Low level mercury area.
- Halide analysis area.
- Dioxin extraction and analysis area
- Hazardous waste storage area
- Oil analysis area

Summit Environmental Technologies, Inc. also maintains a satellite laboratory in Haddock, GA whose work is observed and monitored by the main laboratory in Cuyahoga Falls, OH.

CHAPTER 5

Laboratory Instrumentation and Equipment

5.1 PREVENTIVE MAINTENANCE PROCEDURES AND SCHEDULES

5.1.1 The laboratory maintains detailed logbooks for each instrument documenting the preventive maintenance and repair performed. Maintenance contracts are maintained on all major analytical instruments.

5.1.2 Record keeping of each instrument is the responsibility of the operating technician. This person maintains the instrument logbook detailing how the instrument was used, preventive maintenance performed, and any corrective action undertaken by the analyst.

5.2 CALIBRATION PROCEDURES AND FREQUENCIES

5.2.1 BALANCES

Analytical balances are calibrated daily and before each use, with ASTM Class 2 weights covering the range of its use. Balance calibration checks are documented in a logbook. The balances are serviced and calibrated annually by an ISO 17025 certified company. Calibration weights are calibrated and certified by a certified outside vendor yearly.

5.2.2 OVENS, WATER BATHS, AND INCUBATORS

Oven temperature is checked daily when in use. Thermometers used in ovens are calibrated against a NIST certified thermometer. Water bath and incubator temperatures are checked and recorded during each use.

5.2.3 pH METERS

pH meters are calibrated daily using fresh standard buffer solutions. Buffer solutions are not reused.

5.2.4 INFRARED SPECTROPHOTOMETER

The infrared spectrophotometer calibration is checked with a mid-point calibration check before each use. A failed calibration check requires a new initial calibration.

5.2.5 CONDUCTIVITY METER

The instrument is calibrated against a standard KCl solution each time the instrument is used. The cell constant is determined at least annually.

5.2.6 DISSOLVED OXYGEN METER

The instrument is calibrated according to manufacturer's specifications.

5.2.7 REFRIGERATORS/ FREEZERS

All refrigerators and freezers are monitored for proper temperature by measuring and recording internal temperatures on a daily basis. Refrigerators are maintained at 4 ± 2°C. Freezers are maintained at or below 0°C.

5.2.8 THERMOMETERS

Thermometers used for measurements are calibrated annually against a NIST traceable thermometer.

5.2.9 WATER SUPPLY SYSTEM

The Deionized (DI) water quality is documented on a regular (daily) basis by performing a conductivity and pH test. The DI water meets ASTM Type II standards.

5.2.10 LABORATORY INSTRUMENTATION

Each instrument is calibrated with standard solutions, that are appropriate for the type of instrument and the linear range established for the analytical method. All batches of samples are bracketed by appropriate calibration and verification standards. If calibration checks do not meet established criteria then corrective action is taken that can include recalibration and reanalysis of the affected samples.

5.2.10.1 GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

Each day and prior to the analysis of samples, the instrument is tuned with Bromofluorobenzene (BFB) for volatile compounds and Decafluorotriphenylphosphine (DFTPP) for semi volatile compounds. No samples are analyzed until the instrument has met the tuning criteria of the method. The instrument is then calibrated for each particular method analyses. An initial calibration curve consisting of at least five points is produced to define the working range and to establish criteria and retention time windows for identification. This initial calibration is evaluated every twelve hours to ensure that the system is within calibration and to reestablish the retention time windows. If the daily standard does not meet the established criteria, the system is recalibrated.

5.2.10.2 GAS CHROMATOGRAPHS (GC)

Each chromatographic system is calibrated prior to sample analysis. Initial calibration, which consists of at least five points determines the working range and establishes the retention time windows. The calibration is checked before sample analysis to ensure that the system remains within specification. If the calibration checks do not meet the established criteria, corrective action will be taken including recalibration and reanalysis of the samples.

5.2.10.3 ATOMIC ABSORPTION (AA)

Each instrument is calibrated prior to analysis. A calibration curve is prepared with a minimum of a calibration blank and three standards and then verified with a standard that has been prepared from an independent source at a concentration near the middle of the calibration range. The calibration is then verified on an ongoing basis with a calibration blank and a midpoint calibration standard. If the ongoing calibration standard does not meet established acceptance criteria, corrective action is taken which includes recalibration and reanalysis of samples back to the previous acceptable calibration check. The method of standard additions may be used when matrix interference is present.

5.2.10.4 INDUCTIVELY COUPLED PLASMA (ICP)

The ICP is standardized prior to sample analysis. A curve is prepared with a minimum of a blank and two standards. The curve is then verified with a standard that has been prepared from an independent source at a concentration near the middle of the analytical range. The curve is then verified on an ongoing basis with a blank and a midpoint standard. If the ongoing standard does not meet established acceptance criteria, corrective action is taken which includes re-standardization and reanalysis of samples back to the previous acceptable check.

5.2.10.5 WET CHEMISTRY

This involves a variety of instrument and classical chemistry techniques. Instruments include spectrophotometers, distillation units, and other analyzers. Each system is calibrated before analysis is conducted. Calibration consists of defining the working range by the use of a series of standard solutions. A minimum of five standards is used. The calibration is checked on an ongoing basis (every twenty samples) to ensure that the system remains within specifications. If the ongoing calibration check does not meet established criteria, corrective action is performed which includes recalibration and reanalysis of samples back to the previous acceptable calibration check.

5.2.10.6 SOFTWARE VALIDATION

Instrument software for data management is provided by the instrument manufacturer. The laboratory uses software that has been verified for proper performance by the instrument manufacturer and by comparing instrument results with hand-calculated results. Microsoft ® products are used for creating spreadsheets and word processing documents. Spreadsheet calculations are checked through hand-calculation by the analyst to ensure correct data manipulation.

CHAPTER 6

Document Control

6.1 LABORATORY NOTEBOOK AND LOGBOOK POLICY

6.1.1 All notebooks and logbooks are numbered in a master filing system containing the book number, type, date, and initials of the person to whom issued.

6.1.2 All data entered must be in indelible ink. Errors must not be erased, obliterated, or concealed by correction fluid. Erroneous entries are canceled with a single line drawn through them and initialed and dated by the person making the correction. A brief note may be added to explain the reason for the cancellation.

6.1.3 Blank pages or substantial portions of pages with no entries must be marked with a large "X" or "Z" to indicate that they were deliberately left blank and are not available for addition of new data at a later date.

6.1.4 All entries and corrections in notebooks must be dated and signed by the person making the entry.

6.2 LABORATORY RECORDS MANAGEMENT

6.2.1 Laboratory records are defined as any media that is produced by the laboratory. These include, but are not limited to instrument printouts, logbooks, electronic media, reports, correspondence, memos, invoices, facsimiles, and any other document both written and electronic that is used by the laboratory during the course of business.

6.2.2 Laboratory records are maintained and stored for a minimum of five (5) years from date of origination. The laboratory will maintain and store records for longer periods as requested by the client, agency or governing body. The laboratory records are cataloged and stored in a secure area that is accessible only to laboratory personnel. Storage and disposal records are maintained to monitor document control. The laboratory maintains document control SOPs that give information concerning this process.

CHAPTER 7

Sample Handling and Chain of Custody

7.1 SAMPLE COLLECTION AND HANDLING

Sample collection, handling and preservation techniques are summarized as follows:

Table I
Sample Collection, Handling and Preservation
for Aqueous Matrices

AQUEOUS INORGANICS

Parameter	Preservative	Container	Maximum Holding Time
Acidity	Cool, 4°C	Plastic (P), Glass (G)	14 Days
Alkalinity	Cool, 4°C	P,G	14 Days
Ammonia	Cool, 4°C H_2SO_4 to pH< 2	P,G	28 Days
BOD/CBOD	Cool, 4°C	P,G	24 Hours
Bromide	Cool, 4°C	P, G	28 Days
COD	Cool, 4°C H_2SO_4 to pH < 2	P,G	28 Days
Chloride	None Required	P,G	28 Days
Chlorine, Total Residual	None Required	P,G	Analyze Immediately
Cyanide - Total, Amenable	Cool, 4°C NaOH to pH > 10	P,G	14 Days
Fluoride	None Required	P,G	28 Days
Hardness	HNO_3 to pH < 2	P,G	6 Months
Nitrogen, Kjeldahl	Cool, 4°C H_2SO_4 to pH < 2	P, G	28 Days
Mercury	HNO_3 to pH < 2	P,G	28 Days
Metals	HNO_3 to pH < 2	P,G	6 Months

AQUEOUS INORGANICS
(Cont'd)

Parameter	Preservative	Container	Maximum Holding Time
Nitrate	Cool, 4°C	P, G	48 Hours
Nitrate-Nitrite	Cool, 4°C H_2SO_4 to pH < 2	P, G	28 Days
Nitrite	Cool, 4°C	P, G	48 Hours
Oil and Grease	Cool, 4°C H_2SO_4 to pH < 2	G	28 Days
Organic Carbon	Cool, 4°C H_2SO_4 to pH < 2	P, G	28 Days
Organic Nitrogen	Cool, 4°C	P, G	28 Days
Orthophosphate	Filter immediately Cool, 4°C	P, G	48 Hours
Phenols	Cool, 4°C H_2SO_4 to pH < 2	G	28 Days
Phosphorus	HNO_3 to pH < 2	P, G	28 Days
pH	None required	P, G	Analyze Immediately
Residue, Total	Cool, 4°C	P, G	7 Days
Residue, Filterable (TDS)	Cool, 4°C	P, G	7 Days
Residue, Non-filterable (TSS)	Cool, 4°C	P, G	7 Days
Residue, Volatile	Cool, 4°C	P, G	7 Days
Silica	Cool, 4°C	G	28 Days
Specific Conductance	Cool, 4°C	P, G	28 Days
Sulfate	Cool, 4°C	P, G	28 Days

AQUEOUS INORGANICS
(Cont'd)

Sulfide	Cool, 4°C Zinc Acetate and Sodium Hydroxide	P, G	28 Days
Sulfite	None Required	P, G	48 Hours
Surfactants	Cool, 4°C	P, G	48 Hours
Temperature	None Required	P, G	Analyze Immediately
Total Organic Carbon	Cool, 4°C HCl	P, G	Analyze as soon as possible
Turbidity	Cool, 4°C	P, G	48 Hours

AQUEOUS ORGANICS

Parameter	**Preservative**	**Container**	**Maximum Holding Time**
Volatile Organics*	Cool, 4°C	G, Teflon-lined Septum	14 Days
Extractable Organics**	Cool, 4°C	G, Teflon-lined cap	7 Days until extraction 40 Days after extraction

*Volatile organics include halogenated and non-halogenated compounds, and Gasoline Range Organics. Consult the laboratory prior to sampling for volatile organics to determine the type of sample container and preservative required for sampling.

** Extractable Organics include Base Neutral Acids, Polychlorinated Aromatic Hydrocarbons, PCBs, Pesticides, Herbicides, Diesel Range Organics, Dioxins, and any other compounds that must be extracted from the matrix prior to analysis.

NOTE: Special preservative, containers, and holding time requirements are required for Drinking Water analyses. Please contact the laboratory for information prior to sampling.

Table II
Sample Collection, Handling, and Preservation
for Solid Matrices

SOLID INORGANICS

Parameter	Preservative	Container	Maximum Holding Time
Acidity	Cool, 4°C	Plastic (P) or Glass (G)	14 Days
Alkalinity	Cool, 4°C	P, G	14 Days
Ammonia	Cool, 4°C	P, G	28 Days
Bromide	Cool, 4°C	P, G	28 Days
COD	Cool, 4°C	P,G	28 Days
Chloride	Cool, 4°C	P,G	28 Days
Cyanide, Total Amenable to Chlorination	Cool, 4°C	P,G	14 Days
Fluoride	Cool, 4°C	P,G	28 Days
Nitrogen, Kjeldahl	Cool, 4°C	P, G	28 Days
Mercury	Cool, 4°C	P,G	28 Days
Metals	Cool, 4°C	P,G	6 Months
Nitrate	Cool, 4°C	P, G	48 Hours
Nitrate-Nitrite	Cool, 4°C	P, G	28 Days
Oil and Grease	Cool, 4°C	G	28 Days
Organic Carbon	Cool, 4°C	P, G	28 Days
Orthophosphate	Cool, 4°C	P, G	48 Hours
Phenols	Cool, 4°C	G	28 Days
pH	Cool, 4°C	P, G	Analyze Immediately

SOLID INORGANICS
(cont.)

Silica	Cool, 4°C	G	28 Days
Sulfate	Cool, 4°C	P, G	28 Days
Sulfide	Cool, 4°C	P, G	28 Days
Sulfite	Cool, 4°C	P, G	48 Hours
Surfactants (MBAS)	Cool, 4°C	P, G	48 Hours

SOLID ORGANICS

Parameter	**Preservative**	**Container**	**Maximum Holding Time**
Volatile Organics*	Cool, 4°C	G, Teflon-lined cap	14 Days
Extractable Organics**	Cool, 4°C	G, Teflon-lined cap	14 Days until extraction 40 Days after extraction

*Volatile organics include halogenated and non-halogenated compounds, and Gasoline Range Organics. Consult the laboratory prior to sampling for volatile organics to determine the type of sample container and preservative required for sampling.

** Extractable Organics include Base Neutral Acids, Polychlorinated Aromatic Hydrocarbons, PCBs, Pesticides, Herbicides, Diesel Range Organics, Dioxins, and any other compounds that must be extracted from the matrix prior to analysis.

Table III
Sample Collection, Handling, and Preservation
for Oil Samples

Parameter	**Preservative**	**Container**	**Maximum Holding Time**
All Parameters	None	P, G	None

7.2 SAMPLE LABELING

7.2.1 If requested, the laboratory will supply clients with sample containers (including preservatives if needed) and bottle labels. Samples must be labeled with the following information:

- Client Name.
- Location: The place the sample was taken.
- Time and date when taken.
- Sampler: Name of person who collected the sample.
- Composite or Grab.
- Preservatives used, if any.

7.3 SAMPLE PRESERVATION

7.3.1 The laboratory can provide clients with new and unused sample containers and preservatives. Sample pH is checked and documented for preservatives prior to extraction or analysis. Samples received in unapproved containers will be so noted during sample receipt and log-in.

7.4 SAMPLE LOGIN

7.4.1 Upon arrival, samples are delivered to the sample receiving room. Shipping containers and custody seals are inspected for damage. Samples are unpacked by the sample custodian. Sample identification is verified. Samples are checked according to a "Cooler Receipt Form" for sample receipt conditions. Questions concerning samples are brought to the attention of the laboratory manager and/or clients. The sample is given a unique laboratory number and entered into the Laboratory Information Management System (LIMS).

7.4.2 Once all sample information is obtained, the sample is entered into the LIMS. The LIMS contains the following information:

1. Client name and address
2. Project name
3. Client identification number
4. A unique laboratory identification number
5. Date sampled
6. Time sampled
7. Date and time received
8. Temperature upon arrival
9. Analysis required

7.4.3 The LIMS then tracks the sample from login through to final report generation.

7.4.4 All forms accompanying samples are retained. Chain of custody (COC) forms submitted with samples are kept on file, and a copy of the COC plus the "Cooler Receipt Form" is returned to the client with the Laboratory Report.

7.5 SAMPLE STORAGE

7.5.1 After all sample information is recorded, samples are stored in a walk-in cooler and maintained at 4 ± 2°C. Samples for VOC analysis are segregated and stored in a separate refrigerator in the volatile department at 4 ± 2°C.

7.6 SAMPLE DISPOSAL

7.6.1 After analysis has been completed, the sample is returned to the storage location. The sample is stored for 1-2 months in a cooler and then stored at room temperature for additional 1-2 months prior to disposal. After completion of analyses, samples are disposed of in accordance with state, federal, and local regulations.

7.7 SAMPLE HANDLING

7.7.1 Progress of a sample through the laboratory is summarized as follows:

Sample Received
↓
Sample Inspection – Cooler Receipt Form
Sample entered into LIMS – Sample number assigned
Sample stored in appropriate area
↓
Copy of COC given to Laboratory Manager and staff
↓
Work To Do (WTD) list generated and posted for analysts
↓
Analysis completed
Data reported by analysts
↓
Results reviewed by Laboratory Manager
↓
Data entry
↓
Preliminary report printed
↓
Data reviewed and verified by Laboratory Manager
↓
Final report printed

CHAPTER 8

Analytical Methods

8.1 TEST PROCEDURES

Test procedures used to analyze samples are summarized as follows:

Table 8.1
Classical Chemistry Parameters

PARAMETER	METHOD	DESCRIPTION	REFERENCE
Acidity	SM 2310B	Phenolphthalein endpoint	2
Alkalinity	SM 2320B	Titration	2
Ammonia	SM 4500 NH_3 B, C, E	Distillation/ Nesslerization	2
BOD, 5 Day	SM 5210B	Probe	2
CBOD	SM 5210B	Probe	2
COD	SM 5220B	Colorimetric	2
Bromate	300.1	Colorimetric	1
Bromide	300.0, 300.1	Colorimetric	1,3
Chlorate	300.1	Colorimetric	1
Chlorite	300.1	Colorimetric	1
Chloride	300.0, 9056	Colorimetric	1,3
Chlorine, Residual	SM 4500-Cl G	Colorimetric	2
Cyanide, Total	SM 4500-CN^- E, 9014	Colorimetric Manual Distillation	2,3
Cyanide, Amenable	SM 4500-CN^- G	Colorimetric Manual Distillation	2
Extractable Organic Halides (EOX)	9023	Pyrolysis/ microcoulometry	3

Table 8.1
Classical Chemistry Parameters
(cont.)

Free Liquids	9095A	Free Liquids	3
Flash Point	1010	Pensky-Martens Closed cup	3
Fluoride, Total	300.0, 9056	Colorimetric	1,3
Hardness	200.7, SM 2340C	Titration	1,2
Nitrogen, Kjeldahl	SM 4500-N_{org} B or C	Digestion/ Distillation	2
Nitrate-Nitrogen	300.0, 9056	Colorimetric	1,3
Nitrite-Nitrogen	300.0, 9056	Colorimetric	1,3
Oil and Grease	1664B 9070, 9071A	Gravimetric	1,3
Orthophosphate	300.0, 9056	Colorimetric	1,3
pH	SM 4500-H^+B 9040B, 9045C	Electrometric	2,3
Perchlorate	314.0	Colorimetric	1
Phenolics	SM 5530D	Colorimetric	2
Phosphorus	SM 4500 P E	Colorimetric	2
Reactive Cyanide	Sec 7.3.3.2 SW-846, 3rd Ed.	Gas Evolution	3
Reactive Sulfide	Sec 7.3.4.2 SW-846, 3rd Ed.	Gas Evolution	3
Specific Conductance	SM 2510B, 9050A	Probe	1,2,3
Sulfate	300.0, 9056	Colorimetric	1,2
Sulfide	SM 4500-S^{2-} E 9034	Titration	2,3
Sulfite	SM 4500-SO_3^{2-} B	Titration	2
Surfactants (MBAS)	SM 5540C	Colorimetric	2
Temperature	SM 2550B	Thermometric	2

Table 8.1
Classical Chemistry Parameters
(cont.)

Total Organic Carbon (TOC)	SM 5310B	High Temperature Combustion	2
Total Organic Halides (TOX)	SM 5320B	Pyrolysis/ microcoulometry	2
Turbidity, NTU	180.1, SM 2130B	Nephelometric	1,2
TPH	See Note	Gravimetric	2,3,4
Total Dissolved Solids	SM 2540C	Gravimetric	2
Total Solids	SM 2540B	Gravimetric	2
Total Suspended Solids	SM 2540D	Gravimetric	2
Total Volatile Solids	160.4, SM 2540E	Gravimetric	2,4

Note - Method 418.1 for TPH(IR) is no longer an approved analytical method. Total petroleum hydrocarbons are now determined by Method 1664 or by GC/FID analysis.

Many previous wastewater analytical methods have been deleted by the EPA. These methods are now replaced by analytical methods listed in "American Society for Testing and Materials" (ASTM) and "Standard Methods for the Examination of Water and Wastewater" (SM) 18th - 21st Editions.

Table 8.2
Metal Parameters

Aluminum	200.7, 6010B	ICP	1,3
Antimony	200.7, 6010B	ICP	1,3
Arsenic	7000 Series 200.7, 6010B	AA ICP	1,3
Barium	200.7, 6010B	ICP	1,3
Beryllium	200.7, 6010B	ICP	1,3
Cadmium	200.7, 6010B	ICP	1,3
Calcium	7000 Series 200.7, 6010B	AA ICP	1,3
Corrosivity	SM 2330B	ICP/Calculation	2,3
Chromium	200.7, 6010B	ICP	1,3
Chromium, Hexavalent	7196A	Colorimetric	3
Cobalt	200.7, 6010B	ICP	1,3
Copper	200.7, 6010B	ICP	1,3
Hardness	SM 2340B	ICP/Calculation	2
Iron	200.7, 6010B	ICP	1,3
Lead	3113B, 7421 200.7, 6010B	AA ICP	1,3
Magnesium	200.7, 6010B	ICP	1,3
Manganese	200.7, 6010B	ICP	1,2,3
Mercury	245.1, 7470A, 7471A 1631	Manual – Cold Vapor Low level	1,3
Molybdenum	200.7, 6010B	ICP	1,2,3
Nickel	200.7, 6010B	ICP	1,2,3
Phosphorus	200.7, 6010B	ICP	1,3
Potassium	7000 Series 200.7, 6010B	AA ICP	1,3
Selenium	7000 Series 200.7, 3120B, 6010B	AA ICP	1,3

Table 8.2
Metal Parameters (cont.)

Silicon	200.7, 6010B	ICP	1,2
Silver	200.7, 6010B	ICP	1,3
Sodium	7000 Series 200.7, 6010B	AA ICP	1,2,3
Thallium	7000 Series 200.7, 6010B	AA ICP	1,2,3
Tin	6010B	ICP	1,2,3
Titanium	200.7, 6010B	ICP	1,3
Vanadium	200.7, 6010B	ICP	1,2,3
Zinc	200.7, 6010B	ICP	1,2,3

Table 8.3
Organic Parameters

PARAMETER	METHOD	DESCRIPTION	REFERENCE
Non-Halogenated Volatiles	602, 8015B	GC/FID	3,4
GRO	8015B	GC/FID	3
DRO	8015B	GC/FID	3
PAH	8310 8100 8270C	HPLC GC/FID GC/MS	3
Aromatic Volatiles	602, 8021B	GC/PID	3,4
Volatile Organics	524.2, 624, 8260A,B	GC/MS	3,4,5
Organochlorine Pesticides	508, 608, 8081A	GC/ECD	3,4,5
Organophosphorus Pesticides	614, 8141A	GC/NPD	3,4
Chlorinated Herbicides	515.1, 615, 8151A	GC/ECD	3,4,5
PCBs	508, 608, 8080, 8082	GC/ECD	3,4,5
PVOC	8021B	GC/PID	3
Extractable Organics	525.2, 625, 8270C	GC/MS	3,4,5
Dioxins/Furans	1613, 8290	Hi Res GC/MS	3,4,5
EDB, DBCP, 1,2,3-Trichloropropane	504.1	GC/ECD	5
Endothall	548.1	GC/MS	5
Diquat, Paraquat	549.2	HPLC	5
Haloacetic Acids	552.2	GC/ECD	5
Synthetic Organic Compounds	525.2	GC/MS	5

Table 8.4
Oil Parameters

PARAMETER	METHOD	DESCRIPTION	REFERENCE
Viscosity	ASTM D445	Capillary Viscometer	6
Flash Point	ASTM D92, D93	Pensky Martin	6
Cetane Index	ASTM D1160, D976	Vacuum Distillation	6
% Water	ASTM D6304	Karl Fisher	6
Sulfur & Chlorine	ASTM D4294	XRF	6
BTU	ASTM D240	Bomb Calorimeter	6
Pour Point	ASTM D97	Ice Bath	6
Copper Corrosion	ASTM D130	Water Bath	6
Water and Sediment (BSW)	ASTM D4007	Distillation	6
Color	ASTM D1500	Comparator	6

Table 8.5
Organic Sample Preparation

Description	Method	Technique
TCLP Extraction	SW-846 Method 1311	16-20 Hours Extraction
Liquid/Liquid Extraction	SW-846 Method 3510C	Separatory Funnel
Solid/Liquid Extraction	SW-846 Method 3540C	Soxhlet Extractor
Solid/Liquid Extraction	SW-846 Method 3550B	Sonicator
Waste Sample	SW-846 Method 3580A	Waste Dilution
Volatile Sample	SW-846 Method 5030A&B	Purge and Trap
Volatile Sample	SW-846 Method 5035	Closed System Purge and Trap
Florisil Clean-up	SW-846 Method 3620B	Florisil Column
Silica Gel Clean-up	SW-846 Method 3630C	Silica Gel Column

Table 8.5
Metal Sample Digestion

Description	Method	Technique
Total Recoverable Metals	SW-846 Method 3005A	HCl+HNO_3 Digestion
Total Metals, Aqueous	SW-846 Method 3010A	HNO_3 Digestion
Total Metal, Graphite Furnace	SW-846 Method 3020A	HNO_3 Digestion
Total Metals, Solid	SW-846 Method 3050B	HNO_3 + H_2O_2 Digestion

REFERENCES:

* Federal Register, June 29, 1990

1. "Methods of Chemical Analysis of Water and Wastes". EPA-60014-79-020.

2. "Standard Methods for Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, 18th – 21st Editions.

3. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" USEPA, SW-846, Updates III, IVA, and IVB.

4. "Test Methods for Chemical Analysis of Municipal and Industrial Wastewater." EPA 600/4-82-057, July 1982.

5. "Methods for the Determination of Organic Compounds in Drinking Water" USEPA, July 1990, et. al.

6. "ASTM International" 100 Barr Harbor Drive, West Conshocken, PA, 19428.

CHAPTER 9

Quality System

9.1 QUALITY SYSTEM

9.1.1 Summit Environmental Technologies, Inc. is committed to providing clients with reliable results by maintaining an active quality assurance/quality control program. All laboratory services are performed under a comprehensive quality program that includes written Standard Operating Procedures (SOPs), maintenance logs, data logbooks, employee training and QA/QC sample analysis. Quality audits are regularly scheduled for all analyses performed. The management staff of Summit Environmental Technologies, Inc. oversees all aspects of the laboratory and ensures that the quality system is maintained and enforced. The Quality Control program complies with ISO/IEC 17025 and NELAP guidelines and any federal, state, or local governing bodies.

9.2 QUALITY ASSURANCE/CONTROL

9.2.1 The quality assurance program includes 5-20% duplicate sample analysis, 5-20% laboratory spike samples, and QC check samples. QC check samples are run for every analytical run, and multiple point calibration curves are used. Blank samples are analyzed to verify the absence of contamination. Manual and computer generated charts are used to verify acceptable limits. The laboratory participates in USEPA performance evaluation studies. The laboratory has a testing program to check and rate the performance of each analyst regularly.

9.2.2 Proficiency test samples are used to demonstrate the laboratory performance on a specific set of parameters, methods, or matrices. When unacceptable results are obtained, the problems are investigated, documented, and corrective action is taken.

9.2.3 The laboratory participates in the following proficiency testing programs:

1. DMRQA

2. NELAC Proficiency Testing Program for Wastewater, Solid/Hazardous Wastes, and Drinking Water.

9.3 STANDARD OPERATING PROCEDURES

9.3.1 Standard Operating Procedures (SOPs) are considered as part of the Quality Manual. Due to the number and volume of information contained in the SOPs, it would be cumbersome and impractical to include them in this Quality Manual. The SOPs give a more detailed description of the analytical methods and administrative procedures utilized by the laboratory. The laboratory Quality Manager can be contacted for further information concerning SOP content.

9.3.2 Original copies of current and archived SOPs are kept on file in the Quality Manager's office. Each laboratory section or analyst is given a copy of the SOP that pertains to the procedures required in that particular section. The SOPs are controlled documents that are updated by the Quality Manager as necessary. Copies of pertinent laboratory SOPs are available upon request. All SOPs are under the laboratory's document control procedures.

9.4 DEPARTURES FROM NORMAL PROCEDURES

9.4.1 On occasion, the laboratory must deviate from normal practices and procedures to satisfy a client's analytical or reporting requirements. As a result, certain procedures are in place to notify the client of any departures from established analytical methods or laboratory procedures. Consult the laboratory SOP on Departures for further information.

9.5 COMPLAINTS

9.5.1 During the course of laboratory business, complaints from clients, staff, and other outside sources are received. The complaints are logged and corrective action is initiated to resolve the complaint situation. An SOP is in place to give further instruction in the handling of complaint resolution.

9.6 CONFIDENTIALITY

9.6.1 At certain times, the laboratory may have access to confidential or proprietary information concerning its clients. The laboratory policy is to keep such information confidential and not to reveal any such information to a third party without the written consent of the client. The laboratory has an SOP in place that gives further instruction in this matter.

9.7 LABORATORY CAPACITY

9.7.1 If the laboratory receives samples in excess of the laboratory's analytical capabilities or if the laboratory receives samples for an analysis that is not normally performed by the laboratory, procedures are in place to determine the course of action to be taken in order to both satisfy the client and not jeopardize the quality and accuracy of the sample analysis. An SOP is in place that gives detailed instruction concerning this matter.

9.8 STANDARD/REAGENT PREPARATION PROCEDURES

9.8.1 Primary reference standards and standard solutions used are NIST traceable. All standards and standard solutions are cataloged to identify the supplier, lot number, purity/concentration, receipt/preparation date, technician's name, method of preparation, and expiration data. Standard solutions are validated before use. Stock and working standards are checked regularly for signs of deterioration, discoloration, formation of precipitates, or change in concentration. Standard solutions containers are labeled to identify the chemical(s), concentration, expiration date, technician's initials, and date of preparation. Certificates of analysis are maintained to trace standards that are used for testing purposes. All stock standards must meet a 98% purity standard.

9.8.2 Standard Preparation Logbooks are maintained for the preparation of Calibration Standards which include the following information:

9.8.2.1 The identification number of the concentrated stock standard solution, the date of preparation, the name of analyst who prepared the solution, the chemical compounds in the solution and their lot numbers, the purity, the gross weight, the tare weight, the net weight, the dilution volume, and the concentrations.

9.8.2.2 The identification number of the intermediate concentration standard solution, the date of preparation, the analyst who prepared the solution, the chemical compounds in the concentrated stock, the strength of the concentrated stock, the dilution volume, and the final concentration.

9.8.2.3 The identification number of the working standard solution, the date of preparation, the analyst who prepared the solution, the identification number of the intermediate concentration standards, the dilution volume, and the final concentration.

9.8.2.4 Reagent grade chemicals are used in all tests. All reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society. All standard solutions (stock, calibration, and surrogate) are stored according to supplier's and method recommendations. All stock solutions are replaced after two years or sooner if routine QC indicates a problem with the solution. Stock standards may be used after the expiration date provided that all QC parameters meet acceptance limits. All expired standards are segregated and labeled for disposal.

9.8.2.6 Each calibration standard can be traced back to a certified stock standard through the above procedure. The laboratory maintains the certificates of analysis on file.

9.9 CALIBRATION PROCEDURES AND FREQUENCIES

9.9.1 Non-halogenated Volatiles

This method provides gas chromatographic conditions for the detection of non-halogenated volatile organic compounds. Detection is achieved by Flame Ionization Detector (FID).

Initial Calibration: 5 Levels
Criteria: RSD < 20%
Continuity Daily Calibration: RPD ≤ 15%

9.9.2 Aromatic Volatiles

This method is for the detection of aromatic volatile compounds using GC/PID.

Initial Calibration: 5 levels
Criteria: RSD < 20 %
Continuing Calibration: RPD ≤ 15 %

9.9.3 Pesticides/PCB

This method is for the detection of Pesticides and PCB using GC/ECD. Any compounds identified in the primary analysis will be confirmed on a second GC column.

Initial Calibration: 5 levels
Criteria: RSD < 20 %
Continuing Calibration Check: RPD ≤ 15 %

9.9.4 Volatile Organics

This is a gas chromatography mass spectrometry (GC/MS) method. Identification and quantitation will be performed using response factors and retention times.

Procedure	**Method 624**	**Method 8260A,B**
Tuning	50 ng BFB/Daily	50 ng BFB/ 12 Hours
Initial Calibration	3 Levels	5 Levels
% RSD	< 35	< 15
CCC	Daily	12 Hours
SPCC	Daily	Daily

9.9.5 Semi-volatile Organics

This is a gas chromatography/ mass spectrometry (GC/MS) method. Identification and quantitation will be performed using response factors and retention times.

Procedure	**Method 625**	**Method 8270C**
Tuning	50 ng DFTPP/ Daily	50 ng DFTPP/ 12 Hours
Initial calibration	3 Levels	5 levels
% RSD	< 35	< 15
CCC	Daily	12 Hours
SPCC	Daily	12 Hours

9.9.6 Metals by AA Method 7000 Series

Initial Calibration: Minimum 2 levels
ICV at start of analytical run
CCV every 10 - 20 samples
Criteria: < or + 20% of Expected Value
Correlation Coefficient: Minimum 0.995

9.9.7 Metals by ICP Methods 6010B, 200.7

Initial Standardization: Minimum of 2 levels
ICV at start of analytical run
CCV every 10 – 20 samples
Criteria: < or + 20% of expected value
Correlation Coefficient: Minimum of 0.995

9.9.8 General Chemistry

For Colorimetric Analysis, standard curves used are at least of 5 levels with a minimum correlation coefficient of 0.995. Quality Control Check sample will be analyzed every 20 samples; recovery must be within laboratory established limits.

Refer to the individual analytical SOPs for more complete information on calibration procedures.

9.10 CALIBRATION OUTLIERS

9.10.1 If the value of an initial calibration or calibration verification standard does not fall within the laboratory acceptance limits, the analyst performs corrective actions that may include instrument maintenance such as cleaning the source, changing the column, servicing the purge and trap device, baking out the detector, cleaning the injection port, etc. before proceeding. A new calibration curve is prepared after such corrective action if necessary. Tuning and calibration acceptance criteria must be met before sample analysis can begin. All samples analyzed prior to a failed calibration verification must be reanalyzed back to the most recent compliant calibration verification.

CHAPTER 10

Data Reduction, Validation, and Reporting

All analytical data are reviewed and validated before the final report is generated. This data validation process consists of three levels of documented review.

10.1 TECHNICAL REVIEW LEVELS

10.1.1 ANALYST REVIEW

Each analyst reviews the quality of his/her work based on an established set of guidelines. This review ensures that:

1. Sample preparation information is correct and complete.
2. Analysis information is correct and complete.
3. The appropriate SOPs have been followed.
4. Analytical results are correct and complete
5. QC samples are within established control limits.
6. Blank and laboratory control samples are within appropriate QC limits.
7. Documentation is complete. Level 1 data review is documented by using a check list form that shall be signed and dated by the reviewer.

10.1.2 LABORATORY MANAGER REVIEW

This level is performed by the Laboratory Manager. This review is conducted to ensure that:

1. All appropriate SOPs have been followed.
2. Calibration data are scientifically sound.
3. QC samples are within established guidelines.
4. Quantitative results are correct.
5. Documentation is complete and correct.
6. The data are ready for being incorporated in the final report.
7. The data package is complete on a check list with the signature.
8. This level is documented on a checklist with the signature and date of the reviewer.

10.1.3 DATA QUALITY REVIEW

This level is performed by the Quality Manager. This review is a check of the Laboratory Manger review. It is also documented with the signature and date of the reviewer.

10.2 SAMPLE ANALYSIS

10.2.1 Samples are reanalyzed if any of the following occurs:

a) The sample concentration exceeds the instrument calibration range (samples are diluted).
b) Surrogate recoveries are not within acceptance limits.
c) Method blanks are contaminated.
d) The calibration check is not within acceptance limits.
e) There is a retention time shift.
f) The internal standard responses are not within acceptance limits.
g) The tuning results for GC/MS are not within acceptance limits.
h) There is an instrument malfunction during analysis.

All QC Procedure cited in the gas chromatography methodologies shall be performed and documented.

Gas Chromatography Methods

10.2.2 Quality Control procedures for GC Methods include the following:

- Demonstration of Capability
- Calibration verification
- Method blank analysis
- Analysis of surrogate spiked samples
- Analysis of matrix spike/matrix spike duplicates
- Retention time window check
- Analysis of QA check samples

10.2.3 Quality Control procedures for Gas Chromatograph/Mass Spectrometer Methods

- Demonstration of Capability
- Method blank analysis
- Surrogate Standard spike samples
- Analysis of duplicate samples
- Calibration verification
- Matrix spike duplicate samples
- Daily GC/MS performance tests

10.2.4 Quality Control procedures for Metal Analysis by ICP/AA

- Demonstration of Capability
- Calibration verification
- Analysis of QC check samples
- Method blank analysis
- Matrix spike analysis
- Matrix spike duplicate analysis
- Interference check standard
- Interelement correlation factors

10.2.5 Quality Control procedures for General Chemistry

- Demonstration of Capability
- Check standards
- Blank analysis
- Spike analysis
- Duplicate analysis

10.2.6 Additional Verification

10.2.6.1 Every 10 - 20 samples, depending on method requirements, the laboratory will verify the working standard curve by running an additional standard. Such checks shall be satisfactory only if the results are within ± 10 percent of the expected value.

10.2.6.2 Replicate sample analysis will be conducted for AA and General Chemistry to verify the precision of the method.

10.2.6.3 Samples are spiked at a frequency of 5-20 %, depending on method requirements to verify the accuracy of the method.

10.2.7 Other quality control practices include the following:

- Each analytical balance is checked daily, and records are kept in a logbook.
- A record of the temperature of drying ovens is maintained for each day on which they are in use.
- The accuracy of all thermometers used to monitor temperatures is verified by comparing the readings to a NIST certified thermometer.
- All Quality Control data and records are retained by the laboratory.

10.2.8 General Laboratory Practices

- Analytical reagent grade (AR) chemicals are used for all analyses.
- Laboratory chemicals and reagents, including stock and working standards, are checked regularly for signs of decomposition.
- All reagents are properly labeled with identification of the chemicals, concentration, and date. The name of the analyst is also recorded.
- All chemicals, solutions and standards are dated upon receipt by the laboratory.
- All standards used for instrumental analysis are of high purity
- Compressed gasses used for gas chromatographs are of at least UHP grade.
- All glassware is washed in a warm detergent solution and thoroughly rinsed first with tap water and then with distilled (or deionized water). A separate set of glassware is prepared for dioxin, low level mercury and phosphate analysis.
- Distilled and deionized water shall have minimum resistivity values.

10.3 PROCEDURES USED TO ASSESS PRECISION, ACCURACY, REPRESENTATIVENESS, COMPARABILITY, AND COMPLETENESS

10.3.1 PRECISION

Precision examines the distribution of the reported values about their mean. The distribution of reported values refers to how different the individual reported values are from the average reported values. Precision is a measure of the magnitude of errors that can be measured in a variety of ways. Precision is determined from duplicate samples; thus, precision is expressed as relative percent difference (RPD). Each batch of samples analyzed includes sample duplicates or matrix spikes and matrix spike duplicates to evaluate precision.

10.3.2 RELATIVE PERCENT DIFFERENCE

$$\% \text{ RPD} = \frac{|X1 - X2|}{\frac{(X1 + X2)}{2}} X100$$

Where:
X1, and X2 are the observed measurements

% RPD is used to assess the precision of analyses.

Percent Relative Standard Deviation:

$$\% \text{ RSD} = \frac{\text{Standard Deviation}}{\text{Mean}} X100$$

% RSD is used to assess the precision of a series of analyses.

10.3.3 ACCURACY

Accuracy may be assessed through the use of known and unknown QC samples and spiked samples. Accuracy is usually determined through the use of a Laboratory Control Sample (LCS). Accuracy is presented as percent recovery. Percent bias is a standardized average error; that is, the average error divided by the actual or spiked samples.

10.3.4 PERCENT RECOVERY

$$\%\text{Recovery} = \frac{(SS - SR)}{S} X100$$

Where:
SS = amount measured in spiked sample
SR = amount of sample
S = amount of spike added to the sample

Control charts are established to evaluate precision and accuracy. Control charts are updated periodically. The Western Electric Rules are used for evaluation of control charts. Out-of-control situations are indicated by the following tests:

Test	**Analysis**
1	1 point beyond 3 SD - detects a shift in the process mean, an increase in the estimated standard deviation.
2	9 consecutive points above or below the centerline - detects a shift in the process mean.
3	6 consecutive increasing or decreasing points - detects a trend in process mean.
4	14 consecutive alternating points - detects systematic alternating effects, such as alternating use of different machines, operators or materials.
5	2 or 3 consecutive points beyond 2 SD - detects a shift in the process mean, or an increase in the standard deviation.
6	4 or 5 points beyond 1 SD - detects a shift in the process mean. The 4 points must be on the same side of centerline.
7	15 consecutive points within 1 SD - detects stratification within subgroups when the observations in a single subgroup come from various sources with different means
8	8 consecutive points beyond 1 SD - detects stratification of subgroups.

10.3.5 COMPLETENESS

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions.

Completeness is defined as the percentage of valid results:

%Completeness = A/B X 100

Where:
A = number of valid results
B = total number of possible results.

The laboratory estimate for completeness is 97%.

10.3.6 UNCERTAINTY

Uncertainty is determined by the analysis of LCS samples. Uncertainty is calculated as follows:

$$\text{Uncertainty} = 100 \left(\frac{c}{\overline{R}}\right)\left(1 \pm \frac{L}{\overline{R}}\right)$$

where:

c = concentration of the sample analyte
$\overline{R}$ = the mean LCS recovery associated with the test result
L = the half width of the of the control range (3 SD) for the analyte for a 99% confidence level or the half width of the warning range (2 SD) for the analyte for a 95% confidence level.

Uncertainty confidence levels will be provided to the client upon request.

10.3.7 COMPARABILITY

Summit Environmental Technologies makes every effort to ensure the validity of its analytical data. This policy extends to the integrity of the sample during its collection, preservation and handling, analysis, and reporting of results.
The laboratory uses containers and preservation as recommended in published methods. Analysis begins as soon as possible following the collection of a sample. In order to obtain comparable results, adherence to prescribed standard operating procedures (SOPs) is necessary during sample extraction, analysis, and reporting of results.

10.3.8 REPRESENTATIVENESS

Standard operating procedures are followed that describe sample preparation for solids and liquids to achieve a uniform sample. Sediment samples are mixed thoroughly and foreign objects are discarded before obtaining the sample aliquot. Water samples are allowed to come to ambient temperature and shaken well before taking aliquots for analysis.

10.4 RECORD KEEPING AND DATA REPORTING

10.4.1 After analyses has been completed, a report is generated and accompanied by all raw data, calculations, quality control data for review by the Laboratory Manager. Quality control data are validated according to the following:

- Adherence to quality control elements: spikes, check standards, blanks, duplicates, etc.
- Frequency that these elements were run.
- Comparison of QC results with acceptance criteria.

10.4.2. Records of the analysis, including all raw data, calculations, quality control data, and laboratory reports are kept by the laboratory for a minimum period of five years. Other records are retained as requested by the client or agency.

10.4.3 The following items are also retained by the laboratory:

- The laboratory sample number
- Date, time, and site of sampling
- Name of person who collected the sample
- Date and time of sample receipt by the laboratory
- Date of analysis
- Name of analyst who performed the analysis
- Type of analysis performed and method(s) used
- A copy of the chain of custody

10.5 LABORATORY REPORTS

10.5.1 Laboratory reports include a minimum of the following:

1. A copy of the COC.
2. Date of report
3. Client name and address
4. Date sample collected
5. Date sample received
6. Project name or number
7. Laboratory ID number
8. Client ID number
9. Method of analysis
10. Date of analysis
11. Reporting limits
12. Units
13. Pertinent notes
14. Signature of the Laboratory Manager
15. A copy of the Cooler Receipt Form.

10.6 DATA QUALIFIERS

The laboratory maintains a listing of analytical data qualifiers to indicate any abnormal conditions during sample analysis. The following is a listing of normal data qualifiers:

B = Analyte found in the method blank
J = Analyte concentration between the MDL (LOD) and the Reporting Limit (LOQ). This is considered an "Estimated Concentration".
C = Analyte has been confirmed by another instrument or method
E = Analyte exceeds the upper limit of the calibration curve.
D = Sample or extract was analyzed at a higher dilution
S = Surrogate out of control limits
X = User defined data qualifier. Each "X" qualifier must be defined in the report narrative.

10.7 LABORATORY REPORT SECURITY

In accordance with the requirements of the American Association for Laboratory Accreditation and NELAC, no portion of the laboratory report may be copied or reproduced without the consent of Summit Environmental Technologies, Inc. or its clients. Original copies of all data and ancillary information are retained by the laboratory.

CHAPTER 11

Performance and System Audits

1.1 INTERNAL AUDITS

11.1.1 The Quality Manager is responsible for conducting all internal audits.

11.1.2. Internal audits are conducted on a rotating basis and completed annually.

11.1.3. Checklists covering applicable SOPs and QA/QC aspects are used in conducting audits. These include:

Reagent/method blank analysis
Matrix Spike (MS/MSD) analysis
Surrogate recoveries
Laboratory Control Sample (LCS) recoveries
Calibration standards
Internal standards

11.1.4 Audits include observational assessment of laboratory functions, and the analysis of blind laboratory check samples.

11.1.5 Audit findings are reviewed with personnel audited and laboratory management.

11.1.6 Where corrective action is required, it shall be monitored by the Quality Manager until deficiencies are corrected.

11.1.7 An audit report is prepared documenting observations, corrective actions, and recommendations resulting from the audit. These findings become part of the QA/QC records. The Quality Manager is responsible for maintaining records of all audits and ensuring that corrective action is undertaken.

Audits are conducted to verify that the laboratory operations continue to comply with the requirements of the quality system. The objectives of these audits are to discover:

Whether management objectives are being achieved:
Whether procedures described in the quality system are being followed and opportunities for quality improvements.

11.1.8 Clients are notified if the validity of the analytical results is questionable results as soon as possible after discovery, but no later than 3 days after discovery.

11.2 EXTERNAL AUDITS

11.2.1 External audits are conducted periodically by different state agencies, accrediting agencies, or by clients. External audit review findings and any corrective actions that arise from them are documented.

11.2.2 Existing and prospective clients are permitted to audit the laboratory provided other client confidentiality is maintained.

11.2.3 When deficiencies are noted during an external audit, the laboratory will investigate and correct the deficiency and take steps to prevent future occurrences.

11.3 CORRECTIVE ACTION

11.3.1 When errors, deficiencies, or out-of-control situations exist, the laboratory performs corrective actions to resolve problems and restore proper function to the analytical systems. Personnel are alerted that corrective actions are necessary if:

(1) QC data are outside the acceptable windows for precision and accuracy.

(2) Blanks, duplicate control samples, or control samples contain contamination above acceptable-levels.

(3) Undesirable trends are detected in spike recoveries or RPD between duplicates.

(4) There are unusual changes in method detection limits.

(5) Deficiencies are detected by QA department during internal or external audits or from the results of performance evaluation studies.

Corrective action procedures are initiated at the bench level by the analyst for any obvious analytical or quality control problems. The analyst reviews the preparation or extraction procedure for possible errors. The analyst checks the instruments calibration, spike, and calibration mixes, and instrument sensitivity. If problem persists or cannot be identified, the matter is referred to the Laboratory Manager and QA department for further action. Analytical corrective actions are performed immediately upon discovery. Analysis is stopped until the problem has been resolved. Once the problem is resolved, the corrective action is documented.

11.4 CALIBRATION OUTLIERS

11.4.1 If the value of a check standard does not fall within the acceptance limits, the analyst prepares a new check standard and reanalyzes it. If the new check standard does not fall within the laboratory acceptance limits, the system is inspected for problems. It may be necessary to perform instrument maintenance such as cleaning the source, changing the column, service the purge and trap device, bake out the detector, clean the injection port, etc. before proceeding. A new calibration curve is prepared if necessary. Tuning and/or initial calibration acceptance criteria must be met before sample analysis can begin.

11.4.2 The continuing calibration may not vary from the predicted response by more than ± 10%. Deviation in excess of the specified criteria requires a repeat analysis of each sample analyzed back to the last acceptable check standard. If the continuing calibration acceptance criteria are not met, recalibrate the instrument. Continuing calibration acceptance criteria must be met before any samples are analyzed.

11.5 QUALITY CONTROL OUTLIERS

11.5.1 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

If the spike recovery is out of the spike control limits, the result of the sample may also be suspect. When spike recovery is outside of the specified criteria, this may indicate that either the results obtained for the unspiked sample, that was used for the spike, may not be accurate and the results obtained for all the samples that were analyzed with the unacceptable spike may also be suspect. The following corrective actions are to be used:

a) If an instrument or extraction procedure error was indicated, the spiked sample is re-digested or re-extracted and re-analyzed. If the percent recovery on the reanalysis is within acceptable limits, then the analytical run is considered acceptable.
b) If the percent recovery is outside of acceptance limits, then a matrix effect is assumed.
c) If the percent recovery of a spike is higher than the highest standard used for analysis, then the results are flagged and the LCS results are used.

11.5.2 Surrogate Spikes

Surrogates are added to all samples that are analyzed using GC or GC/MS techniques. They are added prior to purging for volatile compounds and prior to extraction for semi-volatile compounds. Surrogate spike compounds are added to each analytical sample and each QC and calibration sample. Surrogate spike control limits are based on data from past performance. The control limits are updated periodically. If the surrogate recoveries do not meet acceptance criteria, the following corrective actions are necessary:

a) If the surrogate spike recoveries fail acceptance criteria, a new sample is prepared, spiked with surrogate standards and reanalyzed. If the surrogate spike recoveries are within limits, the sample results are considered acceptable.
b) If the surrogate spike recoveries are again out of control limits, then the data is flagged due to matrix interferences.
c) If the sample requires a dilution, and the surrogates are outside of acceptance limits due to dilution, then the surrogate recoveries are flagged as being diluted from the sample.

11.5.3 Internal Standards

If the acceptance criteria for any of the internal standards are not met, then:

a) Check to be sure that there are no errors in standard preparation.
b) Examine the chromatograms for any interfering peaks and integrated peak areas.
c) Check instrument performance. If the instrument performance problem is identified, correct the problem and reanalyze the extract or sample.
d) If no instrument problem is found, then the sample is re-extracted and reanalyzed.
e) If, upon reanalysis, the criteria are not met, then the data is flagged.

11.5.4 Laboratory Control Samples

A LCS is included with each analytical batch. The LCS consists of an aliquot of a clean 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 samples are analyzed using the same procedures employed for the associated samples. The concentration of each analyte in the LCS is calculated against the spike amount to obtain the percent recovery. Samples are related to the LCS recovery by means of a batch ID.

If the LCS recovery is out of control limits, the associated sample results may be suspect. The following corrective actions are used:

a) If the percent recovery is outside of acceptance limits, the analytical system is checked to ensure proper working order and any problems are corrected. The LCS is reanalyzed after any corrections. If the LCS is again out of control limits, all samples associated with the LCS are re-digested or re-extracted and re-analyzed. If the percent recovery on the re-analysis is within acceptable limits, then the analytical run is considered acceptable.

b) If the LCS again fails acceptance limits, all associated sample results are considered suspect and all results are considered "estimated" and indicted as such in the analytical report.

Control limits for LCS are updated semiannually using a minimum of the last 20 or more data points.

11.6 INSTRUMENT MALFUNCTIONS

If an instrument performance problem is identified, then analysis of samples must be halted, and the following performed:

a) Investigate and correct the instrument problem.
b) Recalibrate the instrument if necessary.
c) Reanalyze all samples that were analyzed during the malfunction.

11.7 BLANKS

Two types of analytical blanks are used – Instrument Blanks and Method Blanks.

11.7.1 Instrument Blanks

Instrument blanks are made up of solvent or reagent water with the addition of the identical reagents used in the samples. These blanks are analyzed at a frequency of one every ten samples and at the end of each run. Instrument blanks are considered to be acceptable if they are less than or equal to the reporting limit of the analyte. If this blank exceeds the reporting limit, analysis must cease and the cause of the elevated blank be determined and corrected.

11.7.2 Method Blanks

A method blanks is prepared with each batch of samples. It contains the same reagents as the samples and is prepared by the same procedures. Method blanks are considered to be acceptable if they are less than the detection limit. If a method blank exceeds the reporting limit by more than two times, analysis of the associated samples is halted until the cause is investigated and corrected. Until the source of the contamination is determined, all the samples processed with the method blank are suspect. After the contamination source has been identified and corrected, all samples associated with the contaminated blank must be reprocessed.

Other types of blanks such as trip blanks, field blanks and refrigerator blanks are occasionally analyzed to determine cross contamination of samples while in transit, during sampling and in storage.

CHAPTER 12

Employee Training

12.1 ANALYTICAL METHODS TRAINING

This section applies to employees responsible for sample analysis, as well as data evaluation. Employees are considered trained when they demonstrate that they can run unknown performance evaluation samples and obtain acceptable results. The Ongoing PE sample program is used to track and determine the ability of each analyst to obtain accurate and precise results.

Prior to the issuance a Certification of Competency by the laboratory, the following step-by-step training program must be enacted:

1. Employees will be educated by the Laboratory Manager or department supervisor on the method used for analysis.

2. Employees will receive instruction on proper operation and maintenance of the instrument to be used for analysis. This shall include familiarity with manuals and software.

3. Employees will read the relevant SOP.

4. The Laboratory Manager or department supervisor will educate the employees on the procedure to prepare standards, and will demonstrate to the employees how to integrate all the above steps by analyzing a sample of known concentration.

5. Employees will be educated on the usage and interpretation of chromatogram and computer outputs. Employees will also be shown how to arrive at the final answer by adjusting the computer output by dilution factor, or any other parameter that affects the final results. Employees will be trained on the manual integration rules when manipulating instrument data as specified in the laboratory's Manual Integration SOP.

6. Employees will be shown how to evaluate the final answer, and will be trained on performing QA/QC by determining % Spike recovery, % RPD, and other pertinent parameters.

7. Employees will be allowed to practice by analyzing samples prepared by them until, in the opinion of the Laboratory Manager; they can reasonably operate the instrument.

8. Employees will complete an Initial Demonstration of Capability (IDOC) for each analytical method in their area of responsibility.

12.2 ETHICS TRAINING

12.2.1 It is the policy of Summit Environmental Technologies, Inc. to conduct business with honesty and integrity, and to produce accurate and reliable analytical test results for our clients. As a result, Summit Environmental Technologies, Inc. instructs all of its employees in the understanding that all business conducted by the laboratory is of the highest ethical standard.

12.2.2 No employee is to willfully misrepresent data or records or violate the laws of any federal, state or local institution and is to notify management if such a violation has been observed. Employees violating the ethics policy of the laboratory are subject to disciplinary action, which can include termination or criminal prosecution.

12.2.3 Ethics training refresher courses are conducted for the entire staff on an annual basis. Each employee is given a copy of the laboratory's ethics policy and is required to read and sign a statement that they will comply with the policy.

12.3 TRAINING DOCUMENTATION

12.3.1 Each employee has a "Training Folder" that contains any certificates, diplomas, Demonstrations of Capabilities, or other information that is pertinent to that employee's ongoing training. Employees will attend in-house quality and administrative training sessions on a regular basis as determined by the Quality Manager.

12.3.2 The Laboratory Manager will ensure that each analyst is properly trained in laboratory procedures and analytical methods, and that the analyst is capable of producing accurate and defensible analytical data.

CHAPTER 13

Quality System Review

13.1 Quality System Reviews by management are conducted annually.

13.2 The Quality Manager is responsible for reporting various activities to management staff.

13.3 QA reports to management include:

a) Reports from internal audits.
b) Reports from external lab audits and the laboratory response to external lab audits findings and any corrective action taken.
c) Results of the analysis of blind performance evaluation samples.
d) Results of the analysis of PE samples.
e) Suggestion for purchasing new equipment.
f) Recommendations for additional training of lab personnel.
g) Complaints and reports on any unusual laboratory events, quality assurance anomalies, and corrective actions.
h) Changes in QA/QC procedures.
i) Suitability of policies and procedures.
j) Reports from managerial and supervisory staff concerning laboratory operation.
k) Preventive actions needed or implemented.
l) Changes in the volume or type of work.
m) Client feedback and complaints.
n) Any noncompliance with the rules stated in ISO 17025.

CHAPTER 14

Laboratory Safety

14.1 A Chemical Hygiene Plan is in place and enforced in the laboratory. Safety equipment such as eye wash stations and fire extinguishers are installed and routinely checked to ensure that they are in good working condition. Safety glasses, gloves, face shield, and other protective equipment are available and their use is mandatory whenever applicable.

14.2 Material Safety Data Sheets (MSDS) are available to all employees, and employees receive adequate training by management. To insure that employees are current with S.E.T. safety policies, periodic examinations are given. The employee's score on such tests become part of the employee's record and the company's safety records.

14.3 The laboratory insures that no employee is exposed to health hazards by periodically having its safety policy examined by outside sources. Additionally, periodic air samples are collected over an 8-hr. period to assess exposure levels of employees to chemicals such as Methylene Chloride and Nitric Acid.

14.4 Each employee is instructed in the management of hazardous waste. Hazardous waste is managed and disposed of according to local, state, and federal regulations.

APPENDIX

QC-1
INSTRUMENT PREVENTIVE MAINTENANCE SERVICE

Instrument	Routine Maintenance	Frequency	Major Service
Atomic Absorption	cleaning of instrument cleaning of burner heads replacement of accessories (tubing, graphite tubes, etc.) Cleaning of windows	daily daily as needed monthly	Manufacturer (service contract)
Inductively Coupled Plasma (ICP)	Cleaning of instrument replacement of accessories (tubing)	daily as needed	Manufacturer (service contract)
Gas Chromatography	cleaning of instrument changing septa replacement of columns adjustment of gas flow replacement of liners replacement and cleaning of: FID jet PID lamps	daily after 50 injections as needed weekly High signal Low sensitivity	Manufacturer
Gas Chromatography/Mass Spectrometry	cleaning of instrument cleaning of source filament replacement jet separator cleaning trap replacement check oil level in pump inspect and refill calibration vial change oil in pump Vacuum dust from grills and fans	daily as needed as needed as needed as needed weekly every 3 months every 6 months every year	Manufacturer
High Performance Liquid Chromatography (HPLC)	cleaning of instrument replacement of cartridges replacement of columns	daily as needed as needed	Manufacturer
Infrared Spectrometer	cleaning of instrument clean cell	daily	Manufacturer
UV/Visible Spectrophotometer	cleaning of instrument cleaning of tubes replacement of lamps	after each use after each use as needed	Manufacturer
Pensky-Martens (Flash Point)	cleaning of instrument replacement of tubes	after each use as needed	Manufacturer

Halogen Analyzer	cleaning of instrument cleaning of cell and electrodes	after each use as needed	Manufacturer
Turbidimeter	cleaning of instrument cleaning of tubes replacement of lamp	after each use after each use as needed	Manufacturer
Analytical Balances	Cleaning of balances Yearly calibration	daily	Manufacturer
pH/Ion Meters	cleaning of instruments filling of electrodes solutions replacement of electrodes	after each use as needed as needed	Manufacturer
Ovens	cleaning of oven	as needed	Manufacturer
BOD incubator	cleaning of incubator	as needed	Manufacturer
Water Bath	cleaning of water bath	as needed	Manufacturer
Centrifuge	cleaning of centrifuge	as needed	Manufacturer
Conductance Meter	cleaning of instrument and electrode	after each use	Manufacturer
Sonic Disrupter	cleaning of instrument	after each use	Manufacturer
Evaporators	cleaning of instrument	daily	Manufacturer
Dionex (AS 200)	cleaning of instrument rinsing of transfer lines	daily after each use	Manufacturer
Purge and Trap	conditioning of trap clean glassware with methanol and water/bake	daily-15 minutes after every run	Manufacturer

INSTRUMENT SPARE PARTS

Instrument	Spare Parts
Atomic Absorption	tubing graphite tubes graphite windows
Inductively Coupled Plasma (ICAP)	tubing nebulizer
Gas Chromatography	septa liners FID jets PID lamps reaction tubes seals ferrules autosampler syringes
Gas Chromatography/Mass Spectrometry	filaments source parts jet separator traps
High Performance Liquid Chromatography (HPLC)	cartridges filters
Infrared Spectrometer	lamps cells
UV/Visible Spectrophotometer	tubes lamps
Pensky-Martens (Flash Point)	tubes
Halogen Analyzer	electrodes combustion boats
Turbidimeter	lamps
Conductance Meter	electrode
Evaporators	concentrator tubes
Dionex (AS 200)	transfer lines ferrules

QC-2
METHOD DETECTION LIMIT (MDL)

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99 % confidence that the value is above zero.
Method detection limit is determined for every method the laboratory employs. Method detection limit studies are updated each year.

MDL Determination for Water Samples

1. Prepare seven MDL standards by spiking 7 reagent water samples with 3 to 5 times the concentration of the required detection limit or the lowest analyte concentration used in the initial calibration.

2. Extract these MDL standards following the procedures outlined in the methods SOP.

3. Analyze the MDL standards as described in SOP.

4. Calculate the standard deviation (SD) of the results.

5. Calculate the MDL for each analyte as follows:

MDL = 3.143 X SD

MDL Determination for Solid Samples

1. Prepare seven MDL standards by spiking 3 to 5 times the concentration of the required detection limit or the lowest analyte concentration used in the initial calibration for each analyte into seven sand samples.

2. Extract these MDL standards following the procedures outlined in the method's SOP.

3. Analyze the MDL standards as described in SOP.

4. Calculate the standard deviation (SD) of the results.

5. Calculate the MDL for each analyte as follows:

MDL = 3.143 X SD

6. Adjust for the weight of the samples.

QC-3
QUALITY CONTROL LIMITS CALCULATIONS

1.0 Spike Recovery Calculation

For each matrix spike sample analyzed, calculate the percent recovery of each matrix spike compound added to the sample.

The percent recovery, R, is calculated as follows:

$$\% \text{ Recovery, } R = \frac{(Cs - Cu)}{Cn} X100$$

Where:

Cs = the measured concentration of analyte in the spiked sample aliquot.
Cu = the background concentration of the unspiked sample aliquot.
Cn = the target value, i.e. the known concentration of analyte spiked into the sample aliquot.

The percent recovery data are used to calculate control limits as follows:

a) the percent recoveries and analysis dates of the spiked samples are tabulated,
b) after the analysis of 15-20 matrix spike samples of the same matrix, compute P (the mean percent recovery),
c) compute SD , the standard deviation ,
d) compute WL, the warning limits and CL, the control limits, as follows:

WL = P +/- 2 SD

CL = P +/- 3 SD

Once established, these limits are updated annually as new confidence limits are calculated, using the last 20 points for previous year.

2.0 Surrogate Standard Recovery Calculation

The percent recovery, P, is calculated as follows:

$$\% P = \frac{M}{T} X100$$

Where:

M = the measured value,
T = the target value (i.e. the known value of surrogate spiked into the sample)

A tabulation of percent recoveries is maintained for each surrogate. The tabulation includes the analysis date, the percent recovery and the control limits for P. Warning and control limits are calculated after the analysis of 15-20 samples of the same matrix as follows:

$$WL = P +/- 2 SD$$

$$CL = P +/- 3 SD$$

Where:

WL = the warning limit
CL = the control limit
P = the mean of percent recovery
SD = standard deviation

Once established, these limits are updated annually as new confidence limits are calculated, using the last 20 points for previous year.

3.0 Duplicate Analysis Calculation

The relative percent difference (RPD) is calculated according to the following formula:

$$\% \text{ RPD} = \frac{|C1 - C2|}{\frac{(C1 + C2)}{2}} X100$$

Where:

C1 = Measured concentration of the first sample aliquot
C2 = Measured concentration of the second sample aliquot

Calculate the average (P) and the standard deviation (SD) of % RPD after the analysis of 15-20 matrix spike duplicates of the same matrix.

Calculate warning and control limit as follows:

WL = P +/- 2 SD

CL = P +/- 3SD

Where:

WL = the warning limits
CL = the control limits
P = the mean of % RPD
SD = standard deviation

Once established, these limits are updated annually as new confidence limits are calculated, using the last 20 points for previous year.

QC-4
INTERNAL QUALITY CONTROL CHECKS

Quality control is used to monitor the precision and accuracy of laboratory measurements. Procedures are in place to determine precision and accuracy and these data compared to acceptance criteria established for the analyte and method. Corrective action is taken when the measured precision and accuracy is outside the acceptance criteria.

Quality Control Measures

Quality control measures include the use of check standards, method blanks, instrument blanks, Matrix spike /Matrix spike duplicate samples, Laboratory Control Samples, and surrogate standards.

1.0 Laboratory Check Samples.

1.1 Laboratory check standards are prepared by spiking a known amount of analyte into appropriate solvent/reagent water. For each 12-hour period during which analysis is performed, the performance of the entire analytical system is checked. These checks include the analysis of check standards. Environmental samples are tied to the check standard by means of a date.

1.2 Should a result of a check sample fall outside the acceptance criteria, the analysis is out of control and immediate action is taken to determine the cause of the outlying result. Recalibration of the instrument must take place when the performance changes to the point that the calibration verification acceptance criteria cannot be achieved. Data generated on the same day as the outlying result is regarded as unreliable and the analyses repeated after corrective action has been taken and the procedure is back in control.

2.0 Blanks

2.1 Method blank consists of laboratory -reagent water, which is processed and analyzed as if it was a sample. Method blanks are prepared at a frequency of one method blank for each group of up to 20 samples prepared at the same time, by the same procedures. Samples are related to the laboratory blank by means of date. The blank is calculated as a sample, and blank results for each analyte with the date run and the acceptance criteria maintained.

2.1.1 Acceptance criteria for a method blank must be a result less than or equal to two times the method detection limit.

2.1.2 If the method blank results do not meet the acceptance criteria above, then corrective action is taken to locate and reduce the source of the contamination and to re-extract and reanalyze any samples associated with the contaminated method blank.

2.2 Instrument blanks are analyzed on instruments on which the set of samples were analyzed to demonstrate that the instrument is not contributing contaminants to the samples.

2.2.1 Acceptance criteria for instrument blank must be a result less than the method detection limit.

2.2.2 If the instrument blank results do not meet the acceptance criteria above, then corrective action is taken to locate and reduce the source of the contamination and reanalyze any samples associated with the contaminated instrument blank

3.0 Matrix Spike/Matrix Spike Duplicate, Laboratory Control Samples

3.1 Matrix spike is prepared by dividing the sample into two aliquots. The first aliquot is analyzed as usual. The second aliquot is spiked with a known concentration of the analyte of interest. Prepare a matrix spike duplicate sample from a third aliquot of the sample. One matrix spike/matrix spike duplicate (MS/MSD) pair is extracted with each batch of up to 20 samples of the same matrix processed together. Analyze the MS/MSD samples using the same procedures employed for the original sample, and calculate the concentration of each analyte in the matrix spike and matrix spike duplicate. Samples are related to the spiked recovery by means of a date.

3.2 In addition, a Laboratory Control Sample (LCS) is included with each analytical batch. The LCS consists of an aliquot of a clean 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. Analyze the LCS samples using the same procedures employed for the original sample, and calculate the concentration of each analyte in the LCS. Samples are related to the LCS recovery by means of a date.

3.3 Should spiked recovery result fall outside the acceptance criteria, the analysis is regarded as out of control and immediate action is taken to determine the cause of outlying result. Data generated on the same day as the outlying result is regarded as unreliable and the analysis repeated after corrective action has been taken and the procedure is back in control. Such actions begin with a comparison of the results from the samples or matrix spike samples with the LCS results. If the recoveries of the analytes in the LCS are outside of the control limits, then the problem may lie with the application of the extraction and/or cleanup procedures applied to the sample matrix or with the chromatographic procedures. When the LCS results are within the control limits, the problem may either be related to the specific sample matrix or to an inappropriate choice of extraction, cleanup, and determinative methods.

3.4 Control limits for matrix spike/matrix spike duplicate are updated annually. The last 20 data points for the previous year are used to compute the new limits.

4.0 Surrogate Standard

4.1 A surrogate standard is a compound or a mixture of compounds which, when spiked into environmental sample, behave in the same manner as the analyte or analytes of interest. The surrogate standard has application to organic analytes determined by GC and GC/MS procedures. All samples are spiked with a surrogate standard.

4.1.1 The percent recovery for each surrogate is calculated and compared to acceptance criteria. Should a surrogate standard fall beyond the control limits, the data is regarded as unreliable. Immediate corrective action is taken and the analysis repeated. Corrective actions may include:

4.1.1.1 Check to be sure that there are no errors in the calculations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas.

4.1.1.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the extract (or reanalyze the sample for volatiles).

4.1.1.3 Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the surrogate responses to the point that the recoveries cannot be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required.

4.1.1.4 If no instrument problem is found, the sample is re-extracted and reanalyzed (or reanalyze the sample for volatiles).

4.1.1.5 If, upon reanalysis, the recovery is again not within limits, report the data as an "estimated concentration". If the recovery is within the limits in the reanalysis, use the reanalysis data. If the holding time for the method has expired prior to the reanalysis, provide both the original and reanalysis results to the data user, and note the holding time problem.

4.2 Surrogate standard control limits are updated annually. At least the last 20 data points for the previous year are used to compute new control limits.

QC-5
Reporting Limits

Analysis: GC/MS Volatiles

Parameter	CAS Number	Liquid (ug/L)	Solid (ug/kg)
Benzene	1076-43-3	5.0	5.0
Bromobenzene	108-86-1	5.0	5.0
Bromochloromethane	74-97-5	5.0	5.0
Bromodichloromethane	75-27-4	5.0	5.0
Bromoform	75-25-2	5.0	5.0
Bromomethane	74-83-9	5.0	5.0
n-Butylbenzene	104-51-8	5.0	5.0
sec-Butylbenzene	135-98-8	5.0	5.0
tert-Butylbenzene	98-06-6	5.0	5.0
Carbon Tetrachloride	56-23-5	5.0	5.0
Chlorobenzene	108-90-7	5.0	5.0
Chloroethane	75-00-3	10.0	10.0
Chloroform	67-66-3	5.0	5.0
Chloromethane	74-87-3	10.0	10.0
2-Chlorotoluene	95-49-8	10.0	10.0
4-Chlorotoluene	106-43-4	5.0	5.0
1,2-Dibromo-3-chloropropane	96-12-8	10.0	10.0
Dibromochloromethane	124-48-1	5.0	5.0
1,2-Dibromoethane	106-93-4	5.0	5.0
Dibromomethane	74-95-3	5.0	5.0
1,2-Dichlorobenzene	95-50-1	5.0	5.0
1,3-Dichlorobenzene	541-73-1	5.0	5.0
1,4-Dichlorobenzene	106-46-7	5.0	5.0
Dichlorodifluoromethane	75-71-8	10.0	10.0
1,1-Dichloroethane	75-34-3	5.0	5.0
1,2-Dichloroethane	107-06-2	5.0	5.0
1,1-Dichloroethene	75-35-4	5.0	5.0
cis-1,2-Dichloroethene	156-59-2	5.0	5.0
trans-1,2-Dichloroethene	156-60-5	5.0	5.0
1,2-Dichloropropane	78-87-5	5.0	5.0
1,3-Dichloropropane	142-28-9	5.0	5.0
2,2-Dichloropropane	594-20-7	5.0	5.0
1,1-Dichloropropene	563-58-6	5.0	5.0
Ethylbenzene	1004-41-4	5.0	5.0
Hexachlorobutadiene	87-68-3	5.0	5.0
Isopropylbenzene	98-82-8	5.0	5.0

GC/MS Volatiles
(cont.)

p-Isopropyltoluene	99-87-6	5.0	5.0
Methylene Chloride	75-09-2	5.0	5.0
Naphthalene	91-20-3	5.0	5.0
n-Propylbenzene	103-65-1	5.0	5.0
Styrene	100-42-5	5.0	5.0
1,1,1,2-Tetrachloroethane	630-20-6	5.0	5.0
1,1,2,2-Tetrachloroethane	79-34-5	5.0	5.0
Tetrachloroethene	127-18-4	5.0	5.0
Toluene	108-88-3	5.0	5.0
1,2,3-Trichlorobenzene	87-61-6	5.0	5.0
1,2,4-Trichlorobenzene	120-82-1	5.0	5.0
1,1,1-Trichloroethane	71-55-6	5.0	5.0
1,1,2-Trichloroethane	79-01-6	5.0	5.0
Trichloroethene	79-01-6	5.0	5.0
Trichlorofluoromethane	75-69-4	10.0	10.0
1,2,3-Trichloropropane	96-18-4	5.0	5.0
1,2,4-Trimethylbenzene	95-63-6	5.0	5.0
1,3,5-Trimethylbenzene	108-67-8	5.0	5.0
Vinyl Chloride	75-01-4	2.0	2.0
o-Xylene	95-47-6	5.0	5.0
m,p-Xylene	108-38-3 / 106-42-3	5.0	5.0
2-Butanone	78-93-3	100	100
Acetone	67-64-1	100	100
MIBK	108-10-1	100	100

Analysis: GC/MS Semi-Volatiles

Parameter	CAS Number	Liquid (ug/L)	Solid (mg/kg)
Acenaphthene	83-32-9	10.0	0.2
Acenaphthylene	208-96-8	10.0	0.2
Anthracene	120-12-7	10.0	0.2
Benzidine	92-87-5	50.0	0.5
Benzo[a]anthracene	56-55-3	10.0	0.2
Benzo[a]pyrene	50-32-8	10.0	0.2
Benzo[b]fluoranthene	205-99-2	10.0	0.2
Benzo[g,h,i]perylene	191-24-2	10.0	0.2
Benzo[k]fluoranthene	207-08-9	10.0	0.2
bis (2-Chloroethoxy) methane	111-91-1	10.0	0.2
bis (2-Chloroethyl) ether	111-44-4	10.0	0.2
bis (2-chloroisopropyl) ether	108-60-1	10.0	0.2
bis (2-Ethylhexyl) phthalate	117-81-7	10.0	0.2
4-Bromophenyl phenyl ether	101-55-3	10.0	0.2
Butyl benzyl phthalate	85-68-7	10.0	0.2
2-Chloronaphthalene	91-58-7	10.0	0.2
4-Chlorophenyl phenyl ether	7005-72-3	10.0	0.2
Chrysene	218-01-9	10.0	0.2
Dibenzo[a,h]anthracene	53-70-3	10.0	0.2
1,2-Dichlorobenzene	95-50-1	10.0	0.2
1,3-Dichlorobenzene	541-73-1	10.0	0.2
1,4-dichlorobenzene	106-46-7	10.0	0.2
3,3'-Dichlorobenzidine	91-94-1	50.0	0.2
Diethyl phthalate	84-66-2	10.0	0.2
Dimethyl phthalate	131-11-3	10.0	0.2
Di-n-butyl phthalate	84-74-2	10.0	0.2
2,4-Dinitrotoluene	121-14-2	10.0	0.2
2,6-Dinitrotoluene	606-20-2	10.0	0.2
Di-n-octyl phthalate	117-84-0	10.0	0.2
1,2-Diphenylhydrazine	122-66-7	10.0	0.2
Fluoranthene	206-44-0	10.0	0.2
Fluorene	86-73-7	10.0	0.2
Hexachlorobenzene	118-74-1	10.0	0.2
Hexachlorobutadiene	87-68-3	10.0	0.2
Hexachlorocyclopentadiene	77-47-4	10.0	0.2
Hexachloroethane	67-72-1	10.0	0.2
Indeno[1,2,3-cd]pyrene	193-39-5	10.0	0.2
Isophorone	78-59-1	10.0	0.2

GC/MS Semi-Volatiles
(cont.)

Naphthalene	91-20-3	10.0	0.2
Nitrobenzene	98-95-3	10.0	0.2
N-Nitrosodimethylamine	62-75-9	10.0	0.2
N-Nitrosodi-n-propylamine	621-64-7	10.0	0.2
N-Nitrosodiphenylamine	86-30-6	10.0	0.2
Phenanthrene	85-01-8	10.0	0.2
Pyrene	129-00-0	10.0	0.2
1,2,4-Trichlorobenzene	120-82-1	10.0	0.2
2-Chlorophenol	95-57-8	50.0	0.5
2,4-Dichlorophenol	120-83-2	50.0	0.5
2,4-Dimethylphenol	105-67-9	50.0	0.5
4,6-Dinitro-o-cresol	534-52-1	50.0	0.5
2,4-Dinitrophenol	51-28-5	50.0	0.5
2-Nitrophenol	88-75-5	50.0	0.5
4-Nitrophenol	100-02-7	50.0	0.5
p-Chloro-m-cresol	59-50-7	50.0	0.5
Pentachlorophenol	87-86-5	50.0	0.5
Phenol	108-95-2	50.0	0.5
2,4,5-Trichlorophenol	95-95-4	50.0	0.5
2,4,6-Trichlorophenol	88-06-2	50.0	0.5

Analysis: PAH by HPLC

Parameter	CAS Number	Liquid (ug/L)	Solid (mg/kg)
Acenaphthene	83-32-9	51	1
Acenaphthylene	208-96-8	23	1
Anthracene	120-12-7	11	0.8
Benzo[a]anthracene	56-55-3	12	0.3
Benzo[a] pyrene	50-32-8	10	1
Benzo[b]fluoranthene	205-99-2	10	0.4
Benzo[g,h,i]perylene	191-24-2	9	0.2
Benzo[k]fluoranthene	207-08-9	10	0.3
Chrysene	218-01-9	10	0.9
Dibenzo[a,h]perylene	53-70-3	12	0.3
Fluoranthene	206-44-0	20	0.1
Fluorene	86-73-7	10	0.8
Indeno[1,2,3-cd]pyrene	193-39-5	9	0.2
Naphthalene	91-20-3	20	0.7
Phenanthrene	85-01-8	11	0.9
Pyrene	129-00-0	27	0.8

Analysis: Pesticides

Parameter	CAS Number	Liquid (ug/L)	Solid (mg/Kg)
Aldrin	309-00-2	0.2	0.04
Dieldrin	60-57-1	0.2	0.04
Technical Chlordane	57-74-9	1.0	0.02
4,4'-DDT	50-29-3	0.2	0.04
4,4'-DDE	72-55-9	0.2	0.04
4,4'-DDD	72-54-8	0.2	0.04
Endosulfan I	959-98-8	0.4	0.08
Endosulfan II	33213-65-9	0.4	0.08
Endosulfan sulfate	1031-07-8	0.2	0.04
Endrin	72-20-8	0.2	0.04
Endrin aldehyde	7421-93-4	0.2	0.04
Heptachlor	76-44-8	0.2	0.04
Heptachlor epoxide	1024-57-3	0.2	0.04
alpha-BHC	319-84-6	0.2	0.04
beta-BHC	319-85-7	0.2	0.04
gamma-BHC (Lindane)	58-89-9	0.2	0.04
delta-BHC	319-86-8	0.2	0.04
Toxaphene	8001-35-2	10.0	2.0
Methoxychlor	72-43-5	0.2	0.04

Analysis: PCBs

Parameter	CAS Number	Liquid (ug/L)	Solid (mg/Kg)
Aroclor 1016	12674-11-2	0.2	1.0
Aroclor 1221	11104-28-2	0.2	1.0
Aroclor 1232	11141-16-5	0.2	1.0
Aroclor 1242	53469-21-9	0.2	1.0
Aroclor 1248	12672-29-6	0.2	1.0
Aroclor 1254	11097-69-1	0.2	1.0
Aroclor 1260	11096-82-5	0.2	1.0

Analysis: Miscellaneous Organics

Parameter	Liquid (ug/L)	Solid (mg/kg)
GRO	100	10
DRO	100	10
TPH	1000	10

Parameter	CAS Number	Liquid (mg/L)	Solid (mg/kg)
Aluminum (Al)	7429-90-5	0.05	2.5
Antimony (Sb)	7440-36-0	0.05	2.5
Arsenic (As)	7440-38-2	0.05	2.5
Barium (Ba)	7440-39-3	0.1	5.0
Beryllium (Be)	7440-41-7	0.005	2.5
Cadmium (Cd)	7440-43-9	0.005	0.5
Chromium (Cr)	7440-47-3	0.05	2.5
Cobalt (Co)	7440-48-4	0.05	2.5
Copper (Cu)	7440-50-8	0.05	2.5
Iron (Fe)	7439-89-6	0.05	2.5
Lead (Pb)	7439-92-1	0.1	5.0
Manganese (Mn)	7439-96-5	0.05	2.5
Nickel (Ni)	7440-02-0	0.05	2.5
Phosphorus (P)	7723-14-0	0.5	2.5
Selenium (Se)	7782-49-2	0.05	2.5
Silver (Ag)	7440-22-4	0.05	2.5
Thallium (Tl)	7440-28-0	0.05	2.5
Vanadium (V)	7440-62-2	0.05	2.5
Zinc (Zn)	7440-66-6	0.05	2.5

Analysis: Mercury (Cold Vapor)

Parameter	CAS Number	Liquid (mg/L)	Solid (mg/kg)
Mercury (Hg)	7439-97-6	0.002	0.2

Analysis: Metals (GFAA)

Parameter	CAS Number	Liquid (mg/L)	Solid (mg/kg)
Antimony (Sb)	7440-36-0	0.005	0.25
Arsenic (As)	7440-38-2	0.010	0.25
Lead (Pb)	7439-92-1	0.005	0.5
Selenium (Se)	7782-49-2	0.020	0.5
Thallium (Tl)	7440-28-0	0.002	0.25

NOTE: The above reporting limits are only a partial list of the laboratory's methods and analytes. Consult the laboratory for any additional analysis that may be required. Reporting limits are subject to change without notice due to varying instrument and sample conditions. The above tables are for reference only and give approximate "normal" reporting limit values. Please contact the laboratory for up to date reporting limits for the analytes of interest. Various reporting limits can be achieved to suit the requirements of the client.

Attachment 1-5
Laboratory SOPs

Sections in Analytical SOPs

1. Scope
2. Summary of Method
3. Interferences
4. Estimate of Analytical Time
5. Amount of Sample Required
6. Sampling, Sample Handling, and Preservation
7. Equipment Required
8. Reagents (if applicable)
9. Procedure
10. Quality Control
11. Report
12. Key Words
13. References

WATS SOPs

W-1

Organochlorine & PCB (Arochlor) Pesticide Analysis by GC/ECD

Process Owner (TS/PM/OM/LM) **Randy Eatherton**	
Assignment	Reviewed June 20, 2003 by Christine Devine Reviewed September 11, 2007 by Christine Devine
Electronic only unless colored "Controlled" stamp is present	

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1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentrations of various organochlorine pesticides, and polychlorinated biphenyls (PCBs) as Aroclors, in extracts from municipal and industrial discharges as provided under 40 CRF 136.1. A large number of compounds will give a response in the electron capture detector (ECD) using this method; performance data for the following compounds are provided as part of this method:

ANALYTE	CAS#
alpha-BHC	319-84-6
beta-BHC	319-85-7
delta-BHC	319-86-8
gamma-BHC (Lindane)	58-89-9
Heptachlor	76-44-8
Aldrin	309-00-2
Heptachlor epoxide	1024-57-3
Endosulfan I	959-98-8
Dieldrin	60-57-1
4,4'-DDE	72-55-9
endrin	72-20-8
Endosulfan II	33213-65-9
4,4'-DDD	72-54-8
Endosulfan sulfate	1031-07-8
4,4'-DDT	50-29-3

ANALYTE	CAS#
Methoxychlor	72-43-5
Endrin ketone	53494-70-5
Endrin aldehyde	53494-70-5
alpha-Chlordane	5103-71-9
gamma-Chlordane	5103-74-2
Technical Chlordane	12789-03-6
Toxaphene	8001-35-2
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 This capillary GC/ECD method allows the analyst to use 0.53-mm ID capillary columns (wide bore.)

1.3 Table 1 lists average retention times and Weyerhaeuser method detection limits (MDLs) for each single component analyte of interest for the wide-bore capillary column version of this method. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix and the final volume of the extract. Retention time information given in Table 1 was obtained on two wide-bore, open tubular columns connected to the injector port of a gas chromatograph through a single ferrule.

1.4 Compound identification is based on dual-column/dual-detector analysis. This method describes analytical conditions for a second gas chromatographic column that is used to confirm the measurements made with the primary column. Method 625 (GC/MS) provides additional confirmation on analytes of concentration levels applicable to that method.

1.5 Extracts suitable for analysis by this method may also be analyzed for phthalate esters (606,) Nitroaromatics and Isophorone (609,) Haloethers (611,) and Chlorinated Hydrocarbons (612.)

1.6 This method follows EPA Method 608.

2.0 SUMMARY OF METHOD

2.1 A measured volume of sample, approximately 0.5 Liter, is extracted at neutral pH with methylene chloride using a separatory funnel extraction (AC G-3510.) The extract is dried and exchanged to hexane to a volume of 5 mL (1 mL for lower quantitation limits.) A variety of clean-up steps are applied to the extract, depending on (1) the nature of the co-extracted matrix interferences and (2) the target analytes. Florisil clean up is not mandatory. For samples being analyzed for Aroclors only, sulfuric acid cleanup is applied. If sulfur is detected or suspected, an additional sulfur removal procedure is used. For samples that are suspected of containing oils, GPC (gel permeation chromatography) is performed. After clean up, the extract is analyzed by injecting a 1 to 2-µL sample into a gas chromatograph with dual wide-bore fused silica capillary columns and dual electron capture detectors (GC/ECD.)

2.2 The MDLs achievable in routine analyses of complex samples, using this method, will usually be dependent on the degree of interference associated with the presence of co-eluting EDC-responding compounds rather than on the inherent limitations in detector performance or on the irreducible noise associated with instrument electronics. If interferences prevent identification and qualification of the analytes within quality control (QC) limits at relevant concentrations, this method may also be performed on samples that have undergone clean up. Procedure AC G-3640, Gel-Permeation Clean-up (GPC), is applicable for samples that contain high amounts of lipids, waxes, and other high molecular weight co-extractables.

3.0 INTERFERENCES

3.1 Refer to procedure AP E-8000.

3.2 Sources of interference in this method can be grouped into three broad categories: contaminated solvents, reagents or sample processing hardware; contaminated GC carrier gas, parts, column surfaces or detector surfaces; and the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Knowledge of good laboratory practices is assumed, including steps to be followed in routine testing and clean up of solvents, reagents and sample processing hardware, and instrument maintenance. The discussion following focuses on sources of interference associated with the sample matrix and compound classes that represent common sources of interference, particularly phthalate esters, organosulfur compounds, lipids, and waxes. Interferences co-extracted from the samples will vary considerably from waste to waste. While general clean-up techniques are referenced or provided as part of this method, unique samples may require additional clean-up approaches to achieve desired degree of discrimination and quantitation.

3.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. These materials may be removed prior to analysis using Gel Permeation Cleanup - pesticide option (AC G-3640.) Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive clean up of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

3.4 The presence of elemental sulfur will result in large peaks that interfere with the detection of later eluting organochlorine pesticides. Procedure AC G-3660 is suggested for removal of sulfur, where mercury is used to remove sulfur.

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3.5 Oils, waxes, lipids or other high molecular weight co-extractables can be removed by Gel-Permeation Clean up (AC G-3640.)

3.6 All pesticides extracts are subjected to florisil cartridge clean up (AC G-3620) to remove polar compounds.

3.7 It may be difficult to quantitate Aroclor patterns and single component pesticides together. Pesticides can be removed by sulfuric acid/permanganate clean up (AC G-3665.)

4.0 ESTIMATE OF ANALYTICAL TIME

4.1 Extraction of 1 sample and QC (method blank, Lab control spike, matrix spike and matrix spike duplicate) require about 2 hr (no emulsions formed), 3 hrs (with emulsions).

5.0 AMOUNT OF SAMPLE REQUIRED

5.1 For a sample that requires a matrix spike and duplicate, at least 2 liters of sample is required, otherwise 1 liter of sample is sufficient.

6.0 SAMPLE HANDLING, AND PRESERVATION

6.1 Water samples must be extracted 7 days from date of sampling. Samples are stored under refrigeration (4± 2 °C).

6.2 Extracts must be stored under refrigeration (4± 2 °C) and analyzed within 40 days of extraction.

7.0 EQUIPMENT REQUIRED

7.1 Glassware (see procedure AC G-3510 for requirements.)

7.2 GC vials.

7.3 Kuderna-Danish (K-D) apparatus.

7.4 Gas chromatograph analytical system complete with gas chromatograph (HP5890) suitable for on-column and split/splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detector, and data system (chemstation and chemserver with target). The columns are equipped with 5 m of deactivated 0.53-mm fused silica guard column.

7.4.1 Wide-bore columns

7.4.2 Column 1 - 30 m x 0.53-mm ID fused silica capillary column chemically bonded (Rtx-CLPesticides), 0.50-µm film thickness.

7.4.3 Column 2 – 30 m x 0.53-mm ID fused silica capillary column chemically bonded (Rtx-CLPesticides 2), 0.42-µm film thickness.

7.4.4 Alternate column 1 -30 m x 0.53-mm ID fused silica capillary column chemically bonded with 35 % phenyl methylpolysiloxane (DB 608,) 0.83-µm film thickness.

7.4.5 Alternate column 2 - 30 m x 0.53-mm ID fused silica capillary column chemically bonded with 50 % phenyl methylpolysiloxane (DB 1701,) 1.0-µm film thickness.

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7.4.6 Wide-bore columns are installed in 6.35-mm (1/4-inch) injectors with Siltek Drilled Uniliner (Restek #21055-214.5) deactivated liner designed specifically for use with these columns.

7.4.7 5 meter retention gaps guard column.

7.4.8 De-activated angled glass “Y” connector.

7.5 GC injector ports can be of critical concern, especially in the analysis of DDT and Endrin. Injectors that are contaminated, chemically active, or too hot can cause the degradation ("breakdown") of the analytes. Endrin breaks down to endrin aldehyde and endrin ketone. DDT breaks down to DDD and DDE. Whenever such breakdown is observed, clean and deactivate the injector port (replace or clean liners, replace silicon 'O'-rings, replace bottom of injector port or rinse the whole injector body and silanize), break off at least 5 cm of the guard column (or replace the guard column) and remount it. Check the injector temperature and lower it to 200 °C, if required (200 °C is the minimum operating temperature of the injector). Endrin and DDT breakdown is less of a problem when ambient on-column injectors are used.

8.0 REAGENTS

8.1 Pesticide grade, or equivalent, solvents and reagents shall be used in all tests, unless otherwise indicated. All references to water in this method refer to Millipore water from lab 211S, which shall be organic free.

8.2 Solvents - as appropriate for procedure AC G-3510 - n-hexane, methylene chloride, acetone, and isooctane (2,2,4-trimethylpentane.) Each lot of solvent should be determined to be phthalate free.

DANGER: READ THE HAZARDS FOR SPECIFIC REAGENTS UNDER AP E-3520.

8.3 Stock standard solutions

WARNING: Many of the compounds in the mixed standards are extremely toxic. The practicality of providing hazard evaluation precludes supplying hazard warnings on each substance. Also, the interactive affect of the compounds make providing an overall evaluation impossible. However, most are at very low concentrations. The standards are also handled in a manner such that the likelihood of contact is very small. The greatest **hazard is probably associated with the ability of the solvent used to carry the material through the skin.** There should be little health risks if handled in accordance with safe working practices.

8.3.1 All standards are bought as pre-made solutions that have been tested by the manufacturer. These solutions are then diluted to the required concentration with hexane. The solutions are purchased from Accustandard, Restek, Crescent, and Ultra. Spiking solutions are purchased from different manufacturers than the solutions used for standard curves (i.e., the initial calibration standards). All solutions are purchased with quality control paperwork for the given solutions. Standards are cross- checked by purchasing different standards from different manufacturers (i.e., when individual mix A is replaced, a different source is used for the next new individual mix A).

NOTE: AC Q-StdRefMat for requirements for ordering standards.

8.3.2 Once the vials have been opened, transfer the stock standard solutions into bottles with Teflon-lined screw caps. Store at (4± 2 °C) and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

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8.3.3 Stock standard solutions must be replaced after 1 year (or before the manufacturer's expiration date), or sooner if comparison with check standards indicates a problem.

8.4 Calibration standards:

WARNING: Many of the compounds in the mixed standards are extremely toxic. The practicality of providing hazard evaluation precludes supplying hazard warnings on each substance. Also, the interactive effect of the compounds makes providing an overall evaluation impossible. However, most are at very low concentrations. The standards are also handled in a manner such that the likelihood of contact is very small. The greatest **hazard is probably associated with the ability of the solvent used to carry the material through the skin.** There should be little health risks if handled in accordance with safe working practices.

8.4.1 Calibration standards, at a minimum of five concentrations for each analyte of interest, are prepared through serial dilution of the stock standards with hexane. One of the concentrations should be at a concentration near, but above, the practical detection limit (PQL.) The remaining concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. All calibration solutions must contain the injection standard.

DANGER: All manipulations involving hexane must be performed within a fume hood. n-Hexane is highly flammable. It has a flash point of -23 °C (-9 °F), has explosive limits in air in the range of 1 - 7 %, and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity). n-Hexane can react vigorously with oxidizing materials. Avoid breathing vapors. Exposure can cause dizziness, numbness of extremities, and intoxication. n-Hexane is a central nervous system depressant and neurotoxin. Acute exposure causes irritation, narcosis, and gastrointestinal tract irritation. Chronic inhalation causes peripheral neuropathy and can have neurotoxic effects. Avoid skin contact. n-Hexane is absorbed through the skin. Prolonged or repeated skin contact can cause irritation and dermatitis, through defatting of skin.

8.4.2 Calibration solutions must be replaced after 1 year (or before the stock expiration date) or sooner, if comparison with check standards indicates a problem.

8.4.3 Two calibration mixtures are prepared (by serial dilution of a purchased stock standard) for the single component analytes (i.e., Individual Mix A and B) of this method to eliminate potential resolution and quantitation problems.

8.5 Internal standards

The analyst selects one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Isodrin is used as the internal standard. This internal standard is used to check the injection of the sample. All of the calibration standards, instrument blanks and samples have this added. The peak height must be within 80 to 120 % recovery for the injection to be deemed as a good injection. Poor injection indicates that the Teflon plug on the syringe plunger may need to be replaced, that the needle is partially plugged, or that the sample is interfering with the internal standard.

8.6 Surrogate standards

The analyst should monitor the performance of the extraction, clean up (when used), and analytical system, and the effectiveness of the method in dealing with each sample matrix, by spiking each

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sample, standard, and water blank with pesticide surrogates. Because GC/ECD analyses are more subject to interference than GC/MS analysis, a secondary surrogate is to be used when sample interference is apparent. Decachlorobiphenyl is the primary surrogate, and should be used whenever possible. However, if recovery is low, or compounds interfere with decachlorobiphenyl, then 2,4,5,6-tetrachloro-m-xylene should be evaluated for acceptance. Proceed with corrective action when both surrogates are out of limits for a sample (part 10.3.1). Surrogate solutions are purchased at the concentration of 200µg/mL. This solution is diluted to 0.2 µg/mL in methanol.

9.0 PROCEDURE

9.1 Extraction:

9.1.1 In general, water samples are extracted with methylene chloride as a solvent using a separatory funnel extraction (procedure AC G-3510.)

9.1.2 Spiked samples (Matrix Spikes or MSs) and spiked blanks (Lab Control Spikes or LCSs) are used to verify the applicability of the chosen extraction technique to each new sample type. The spiked sample must be spiked with the compounds of interest to determine the percent recovery and the limit of detection for that sample.

Spiking of water samples should be performed by adding appropriate amounts of pesticide or PCB compounds, dissolved in methanol, to the water sample immediately prior to extraction. After addition of the spike, mix the samples manually for 1 to 2 min. Typical, spiking concentrations for water samples are: 0.5 to 1.0 µg/L for samples in which pesticides and PCBs were not detected; and 2 to 5 times the background concentration in those cases where pesticides and PCBs are present (use of mixtures of Aroclors other than 1016/1260 are <u>not recommended</u> with this method.) Weyerhaeuser uses the submitter's specified samples for spiking. If this is not available from the submitter, then the lab chooses at random which sample will be used for matrix spiking.

9.2 Clean up/Fractionation

9.2.1 Clean-up 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 clean-up 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 clean up is provided in this part.

9.2.2 If a sample is suspected of containing oils or other high molecular compounds, the use of GPC clean up/pesticide option (AC G-3640) is followed by florisil cartridge clean up (AC G-3620.)

9.2.3 If only PCBs are to be measured in a sample, the sample is subjected to sulfuric acid clean up (AP E-3665, where only the sulfuric acid clean up is employed), followed by Florisil cartridge clean up (AC G-3620).

9.2.4 If both PCBs and pesticides are to be measured in the sample, isolation of the PCB fraction by silica gel fractionation (procedure AC G-3630) is recommended. This procedure has not been tested in the extraction lab and is only mentioned as an option for later development.

9.2.5 If only pesticides are to be measured, clean up by AC G-3620 is used.

9.2.6 Elemental sulfur, which may appear in certain industrial wastes, interferes with the electron capture gas chromatography of certain pesticides. Sulfur should be removed by the technique described in AC G-3660, Sulfur Clean up, using mercury.

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9.3 Gas chromatography conditions (recommended):

Wide-bore columns (DB1701 and DB608):	
Carrier gas (He)	5-7 mL/min
Makeup gas (argon / 5 % methane)	100-150 mL/min
Injector temperature	200 °C
Detector temperature	320 °C
Initial temperature	110 °C, hold 0.5 min
Temperature program	110 °C to 270 °C at 9 °C/min
Final temperature	270 °C, hold 10 min.

Wide-bore columns (Rtx-CLPestides and Rtx-CLPesticides 2):	
Carrier gas (He)	18 mL/min
Makeup gas (argon / 5 % methane)	100-150 mL/min
Injector temperature	200 °C
Detector temperature	300 °C
Initial temperature	110 °C, hold 1.0 min
Temperature program	110 °C to 200 °C at 8 °C/min 200 °C to 270 °C at 8 °C/min
Final temperature	270 °C, hold 4.75 min.

9.4 Calibration:

9.4.1 Refer to AI G-8000 for proper calibration techniques. Table 2 is used for the lowest point on the calibration curve (i.e., the low standard is used for the quantitation limits.) If the laboratory is requested to use MDL, then Table 1 is used for the quantitation limits.

9.4.2 The procedure for internal calibration is used. Refer to AI G-8000 for a description for this procedure.

9.4.3 Two calibration mixtures are used in order to minimize the problem of co-elution. These calibration mixtures are also listed in Table 2, along with the low point concentration of each analyte in the mixture. The concentrations provided should be detectable on a GC/ECD suitable for use with this method. Mixtures of Aroclors other than 1016/1260 are not recommended for use with this method (i.e., all of the other Aroclors are individually analyzed.)

9.5 Gas chromatographic analysis:

9.5.1 Refer to AI G-8000. Add 10 µL of internal standard (i.e., injection standard) to the sample extract prior to injection.

9.5.2 Follow AI G-8000 for instruction on the analysis sequence, appropriate dilutions, establishing daily retention time windows, identification criteria, and analysis of a mid-concentration standard after each group of 10 samples.

9.5.3 Examples of GC/ECD chromatograms generated by instruments with wide-bore columns are presented in Figures 1 through 10.

9.5.4 The sample volume injected and the resulting peak sizes (in peak heights) are recorded via the chemstation software and transferred to the chemserver for processing via target software.

9.5.5 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine

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whether further concentration of the sample is warranted by the context in which the result is to be used. In reporting, these values will be 'J' flagged (detected but quantitation is only an estimate.)

9.5.6 If the peak response exceeds the working range of the system, dilute the extract and re-analyze. In reporting, these values will be 'E' flagged (detected at a value greater than the calibration curve and the value is only an estimate). Dilute the samples so that the response is at the mid calibration level.

9.5.7 Identification of mixtures (i.e., PCBs, technical chlordane and toxaphene) are based on the characteristic "fingerprint" retention time and shape of the indicator peak(s). Quantitation is based on the height of the characteristic peaks as compared to the height of the corresponding calibration peak(s) of the same retention time and shape generated using external calibration procedures (AI G-8000, 7.6.a). At least three peaks are used in the quantitation of multicomponent analytes.

9.5.8 Identify compounds in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. Due to the stability of megabore columns, the window will never be greater than 0.07 min.

9.5.9 Quantitation of the compound(s) of interest is premised on: 1) a linear response of the ECD to the ranges of concentrations of the compound(s) of interest encountered in the sample extract and the corresponding calibration extract; and 2) a direct linear proportionality between the magnitude of response of the ECD over the width(s) of the retention window(s) (the height of the characteristic or "fingerprint" peak[s]) in the sample and calibration extracts. Proper quantitation requires the appropriate selection of a baseline from which the height of the characteristic peak(s) can be calculated.

9.5.10 If compound identification or quantitation are precluded due to interference (e.g., broad, rounded peaks or ill-defined baseline are present) clean up or dilution of the extract is warranted. Running of instrument blanks followed by midpoint calibration checks are used to determine if the instruments integrity has been affected by the sample is necessary before further sample analysis can be continued. Refer to part 3.2 for the procedures to be followed in sample clean up. If a sample is suspected of being high concentration or high background, multiple instrument blanks are added after the sample. While these instrument blanks are not analyzed, they serve visually to demonstrate that there is no carryover into the next analyzed sample.

9.6 Quantitation of Multiple Component Analytes:

Scope (excerpted from U.S. FDA, PALM): Residues of mixtures of two or more components present problems in measurement. When they are found together, e.g., toxaphene and DDT, the problem of quantitation becomes even more difficult. The following parts are for handing quantitation of toxaphene, chlordane, PCB, DDT, and BHC.

9.6.1 Toxaphene: Quantitative calculation of toxaphene or Storbane is difficult, but reasonable accuracy can be obtained. To calculate toxaphene on GC/ECD, (a) adjust the sample size so that the major toxaphene peaks are 10 - 70 % of full-scale deflection (FSD); (b) inject a toxaphene standard that is estimated to be within ± 10 ng of the sample; and (c) quantitate using total area sum or sum of all peaks in the range of the standard. This procedure is made difficult by the fact that the relative heights and widths of the peaks in the sample will probably not be identical to the standard.

9.6.2 Chlordane is a technical mixture of at least 11 major components and 30, or more, minor components. Alpha and gamma chlordane are the two major components of technical chlordane. However, the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch.

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a. The GC pattern of a chlordane residue may differ considerably from that of the technical standard. Depending on the sample substrate and its history, residues of chlordane can consist of almost any combination of: constituents from the technical chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight.

b. When the chlordane residue does not resemble technical chlordane, but instead consists primarily of individual, identifiable peaks, quantitate the peaks of alpha-chlordane, gamma-chlordane, and heptachlor separately against the appropriate reference materials, and report the individual residues.

c. When the GC pattern of the residue resembles technical chlordane, the analyst may quantitate chlordane residues by comparing the height of the chlordane-chromatogram using the three major peaks (alpha and gamma chlordane and heptachlor). If heptachlor and/or heptachlor epoxide are much out of proportion, calculate these separately and subtract their height from the total area to give a corrected chlordane height. (Note that octachlor epoxide, a metabolite of chlordane, can easily be mistaken for heptachlor epoxide on a nonpolar GC column.)

d. To measure the height of the chlordane chromatogram, proceed as in toxaphene. Adjust the sample volume to produce a response in which alpha and gamma chlordane are approximately the same size as those in the initial calibration standard. Quantitate on at least three peaks.

9.6.3 Polychlorinated biphenyls (PCBs): Quantitation of residues of PCB involves problems similar to those encountered in the quantitation of toxaphene, strobane, and chlordane. In each case, the chemical is made up of numerous compounds and so the chromatograms are multi-peak. Also, in each case, the chromatogram of the residue may not match that of the standard.

a. Mixtures of PCB of various chlorine contents were sold for many years in the U.S. by the Monsanto Co. under the tradename Aroclor (1200 series and 1016). Though these Aroclors are no longer marketed, the PCBs remain in the environment and are sometimes found as residues in foods, especially fish.

b. PCB residues are quantitated by comparison to one or more of the Aroclor materials, depending on the chromatographic pattern of the residue. A choice must be made as to which Aroclor, or mixture of Aroclors, will produce a chromatogram most similar to that of the residue. This may also involve a judgement about what proportion of the different Aroclors to combine to produce the appropriate reference material.

c. Quantitate PCB residues by comparing height of residue peaks to height of peaks from appropriate Aroclor(s) reference materials. Measure height response from common baseline under all peaks. Use only those peaks from sample that can be attributed to chlorobiphenyls. These peaks must also be present in the chromatogram of reference materials. Mixture of Aroclors may be required to provide best GC patterns of sample and reference.

9.6.4 DDT: DDT found in samples often consist of both o,p' and p,p' DDT. Residues of DDE and TDE are also frequently present. Each isomer of DDT and its metabolites should be quantitated using the pure standard of that compound and reported as such. Only the para para' isomers are quantitated in the lab.

9.6.5 Hexachlorocyclohexane (BHC, from the former name, benzene hexachloride): Technical grade BHC is a cream-colored amorphous solid with a very characteristic musty odor. It consists of a mixture of six chemically distinct isomers and one or more heptachloro-cyclohexanes and octachloro-cyclohexanes.

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a. Commercial BHC preparations may show a wide variance in the percentage of individual isomers present. The elimination rate of the isomers fed to rats was 3 weeks for the alpha, gamma and delta isomers and 14 weeks for the beta isomers. Thus it may be possible to have any combination of these various isomers in different food commodities. BHC found in dairy products usually has a large percentage of beta isomer.

b. Alpha, gamma, and delta isomers show equal response on ECD, whereas beta isomer shows a much weaker electron affinity compared to the other isomers.

c. Quantitate each isomer separately against a standard of the respective pure isomer.

10.0 QUALITY CONTROL

10.1 Quality control to validate sample extraction is covered in AC G-3500 and in the extraction method utilized. If extract clean up is performed, follow the QC in AC G-3600 and in the specific clean-up method.

10.2 Mandatory quality control to evaluate the GC system operation is found in AI G-8000.

10.2.1 The quality control check sample concentrate (AI G-8000) contains each single-component parameter of interest at the following concentrations (withing 5% of the stated value) in acetone: P,P' DDD, P,P' DDT, Endosulfan II, Endosulfan Sulfate and Endrin at 1.0 µg/mL; and the rest of the single component pesticides at 0.20 µg/mL. If this method is only to be used to analyze PCBs, technical Chlordane or Toxaphene, the QC check sample concentrate should contain the most representative multi-component parameter at a concentration of 5.0 µg/mL.

10.2.2. Table 3 indicates the EPA's calibration and QC acceptance criteria for this method. Table 4 gives the EPA's method accuracy and precision as functions of concentration for the analytes of interest. The contents of the Tables are used to evaluate the laboratory's ability to perform and generate acceptable data by this method.

10.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits (limits established by performing QC procedures outlined in AI G-8000).

10.3.1 If recovery is not within limits, the following are required:

a. Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

b. Recalculate the data and/or re-analyze the extract if any of the above checks reveal a problem.

c. Re-extract and re-analyze the sample if none of the above are a problem or flag the data as "estimated concentration."

10.3.2. The breakdown of DDT and endrin should be measured before samples are analyzed. Injector maintenance and re-calibration should be completed if the breakdown is greater than 20 % for either compound. If neither of these analytes is detected in the sample, then this breakdown is noted but does not invalidate the data.

10.3.3. Include an instrument blank followed by mid-concentration calibration standard after each group of 10 samples in the analysis sequence as a calibration check. The response factors for the mid-concentration calibration should be within 20 percent of the average values for the multi-concentration calibration. When this continuing calibration is out of this acceptance window, the laboratory should stop analyses, clean the injector and replace the septum. An instrument blank

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and the continuing calibration standard are analyzed again to determine if the system needs to be re-calibrated. Analysis may be continued if the response factor is within the 20 % value. Otherwise, the system must be re-calibrated.

10.3.4. Internal standards must be evaluated for acceptance. The measured height of the internal standard must be no more than 20 % different from the height from the mid- calibration. When the internal standard peak height is outside the limit, the problem must be corrected before any further analysis is done. Samples that fall outside the QC criteria must be re-analyzed.

10.4 GC/MS confirmation: Any compounds confirmed by two columns should also be confirmed by GC/MS if the concentration is sufficient for detection by GC/MS as determined by the laboratory generated detection limits.

10.4.1 The GC/MS would normally require a minimum concentration of 10 ng/L in the final extract for each single-component compound.

10.4.2 The pesticide extract and associated blank should be analyzed by GC/MS as Method AM E-8270.

10.4.3 The confirmation may be from the GC/MS analysis of the base/neutral-acid extracts (sample and blank). However, if the compounds are not detected in the base/neutral-acid extract, even though the concentrations are high enough, a GC/MS analysis of the pesticide extract should be performed.

10.4.4 A QC reference sample of the compound must also be analyzed by GC/MS. The concentration of the QC reference standard must demonstrate the ability to confirm the pesticides/Aroclors identified by GC/ECD.

10.5 METHOD PERFORMANCE

10.5.1 The MDL concentrations listed in Table 1 were obtained using organic-free reagent water. The MDL actually achievable in a given analysis will vary depending on detector response characteristics, irreducible noise from instrument electronics and matrix effects. The single component pesticides along with aroclor 1242 are represented in this table.

10.5.2 Following this method, the obtainable accuracy and precision will be determined by the sample matrix, sample preparation technique, optional clean-up techniques, and the calibration procedures used. The supplied accuracy and precision for water analysis from the EPA are listed in Table 4.

11.0 REPORT

11.1 CALCULATIONS: [(µg/mL analyte/respose factor) x mL final volume x dilution factor] /amount extracted (in L for liquids)

11.2 PRECISION AND ACCURACY. See 10.5.2.

11.2.1 Units for reporting are µg/L (ppb).

11.2.2 Report all concentrations above the quantitation limits to 2 significant figures. Report values below the quantitation limit to 1 significant figure.

12.0 KEY WORDS

Proprietary — Disclosure limited to persons confidentially bound to Weyerhaeuser.

AM E-608, Arochlor, ECD, electron capture, EPA, Kuderna-Danish, KD, K-D, organochlorine, PCB, pesticide, polychlorinated biphenyls

13.0 REFERENCES

13.1 Code of Federal Regulations 40, Part 136, Appendix A, Method 608.

13.3 Organics Analysis - Multi-Media, Multi-Concentration, Document OLM 1.6, June 1991.

14.0 REVISION HISTORY

14.1 9/11/07 - Updated the codes to the referenced SOPs.

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TABLES

TABLE 1
Weyerhaeuser's Annual Established Method Detection Limit (MDL) with retention times

ANALYTE	RT - DB1701	RT- DB608	µg/L 2/95 WATER DB1701	µg/L 2/95 WATER DB608
alpha-BHC	6.85	9.48	0.0008	0.0020
beta-BHC	8.28	13.23	0.0005	0.0019
delta-BHC	9.42	13.98	0.0016	0.0022
gamma-BHC	8.04	10.77	0.0006	0.0012
Heptachlor	9.03	11.40	0.0006	0.0017
Aldrin	10.13	12.28	0.0003	0.0025
Heptachlor_epoxide	11.90	14.53	0.0008	0.0018
Endosulfan_I	13.02	15.42	0.0004	0.0020
Dieldrin	14.04	16.58	0.0007	0.0021
4,4'-DDE	13.84	16.01	0.0006	0.0043
endrin	15.23	17.28	0.0007	0.0029
Endosulfan_II	15.84	18.82	0.0006	0.0043
4,4'-DDD	15.67	18.55	0.0007	0.0024
Endosulfan_sulfate	17.35	21.62	0.0008	0.033
4,4'-DDT	16.63	19.07	0.0011	0.0034
Methoxychlor	19.54	21.32	0.0036	0.0024
Endrin_ketone	19.90	23.48	0.0013	0.0028
Endrin_aldehyde	16.92	20.33	0.0020	0.0031
alpha-Chlordane	12.97	15.79	0.0004	0.0023
gamma-Chlordane	12.43	15.59	0.0004	0.0019
Chlordane (technical)	*	*	0.0047	0.0059
Toxaphene	*	*	0.10	0.11
Aroclor 1016	*	*	ND	ND
Aroclor 1221	*	*	ND	ND
Aroclor 1232	*	*	ND	ND
Aroclor 1242	*	*	0.016	0.012
Aroclor 1248	*	*	ND	ND
Aroclor 1254	*	*	ND	ND
Aroclor 1260	*	*	ND	ND

e = Multiple peak response
ND = not determined

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TABLE 2
Low Concentration of the Individual Component Mixes

INDIVIDUAL MIX A	µg/mL
alpha-BHC	0.005
gamma-BHC	0.005
Heptachlor	0.010
Endosulfan_I	0.010
Dieldrin	0.010
endrin	0.010
4,4'-DDD	0.010
4,4'-DDT	0.010
Methoxychlor	0.050
INDIVIDUAL MIX B	µg/mL
Beta BHC	0.005
Delta BHC	0.005
Aldrin	0.005
Heptachlor Epoxide	0.005
alpha Chlordane	0.005
gamma Chlordane	0.005
4,4'-DDE	0.010
Endosulfan II	0.010
Endrin Aldehyde	0.010
Endosulfan Sulfate	0.010
Endrin Ketone	0.010
SEPARATE MULT- COMPONENT ANALYTES	µg/mL
Aroclor 1016/1260	0.100
Aroclor 1221	0.200
Aroclor 1232	0.100
Aroclor 1242	0.100
Aroclor 1254	0.100
Technical Grade Chlordane	0.050
Toxaphene	0.500

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TABLE 3
EPA QC ACCEPTANCE CRITERIA FOR WATER ANALYSIS

Analyte	Test Conc. (µg/L)	limit for s (µg/L)	Range P,Ps (%)	Range of $\bar{x}$ (µg/L)
Aldrin	2.0	0.420	42 - 122	1.08 - 2.24
alpha BHC	2.0	0.480	37 - 134	0.98 - 2.44
beta BHC	2.0	0.640	17 - 147	0.78 - 2.60
gamma BHC	2.0	0.720	19 - 140	1.01 - 2.37
delta BHC	2.0	0.460	32 - 127	0.86 - 2.32
Technical Chlordane	50.0	10.0	45 - 119	27.6 - 54.3
DDE	2.0	2.80	31 - 141	4.80 - 12.6
DDD	10.0	0.55	30 - 145	1.08 - 2.60
DDT	10.0	3.60	25 - 160	4.60 - 13.7
Dieldrin	2.0	0.760	36 - 146	1.15 - 2.49
Endosulfan I	2.0	0.490	45 - 153	1.14 - 2.82
Endosulfan II	10.0	6.10	d - 202	2.20 - 17.1
Endosulfan Sulfate	10.0	2.70	26 - 144	3.80 - 13.2
Endrin	10.0	3.70	30 - 147	5.10 - 12.6
Heptachlor	2.0	0.040	34 - 111	0.86 - 2.00
Heptachlor Epoxide	2.0	0.410	37 - 142	1.13 - 2.63
Toxaphene	50.0	12.7	41 - 126	27.8 - 55.6
Aroclor 1016	50.0	10.0	50 - 114	30.5 - 51.5
Aroclor 1221	50.0	24.4	15 - 178	22.1 - 75.2
Aroclor 1232	50.0	17.9	10 - 215	14.0 - 98.5
Aroclor 1242	50.0	12.2	39 - 150	24.8 - 69.6
Aroclor 1248	50.0	15.9	38 - 158	29.0 - 70.2
Aroclor 1254	50.0	13.8	29 - 131	22.2 - 57.9
Aroclor 1260	50.0	10.4	8 - 127	18.7 - 54.9

s= Standard Deviation of four recovery measurements, in µg/L.
$\bar{x}$ = Average recovery for four recovery measurements, in µg/L.
P, Ps = Percent recovery measured
D = Detected; result must be greater than zero.

Criteria from 40 CFR Part 136 for Method 608. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

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TABLE 4
EPA METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATIONS

Analyte	Accuracy, as recovery, x' (µg/L)	Single Analyst precision, s' (µg/L)	Overall precison, S' (µg/L)
Aldrin	0.81C+0.04	0.16x-0.04	0.20x-0.01
alpha BHC	0.84C+0.03	0.13x+0.04	0.23x+0.00
beta BHC	0.81C+0.07	0.22x+0.02	0.33x-0.95
gamma BHC	0.81C+0.07	0.18x+0.09	0.25x+0.03
delta BHC	0.82C-0.05	0.12x+0.06	0.22x+0.04
Technical Chlordane	0.82C-0.04	0.13x+0.13	0.18x+0.18
DDE	0.85C+0.14	0.20x-0.18	0.27x-0.14
DDD	0.84C+0.30	0.13x+0.06	0.28x-0.09
DDT	0.93C-0.13	0.17x+0.39	0.31x-0.21
Dieldrin	0.90C+0.02	0.12x+0.19	0.16x+0.16
Endosulfan I	0.97C+0.04	0.10x+0.07	0.18x+0.08
Endosulfan II	0.93C+0.34	0.41x-0.65	0.47x-0.20
Endosulfan Sulfate	0.89C-0.37	0.13x+0.33	0.24x+0.35
Endrin	0.89C-0.04	0.20x+0.25	0.24x+0.25
Heptachlor	0.69C+0.04	0.06x+0.13	0.16x+0.08
Heptachlor Epoxide	0.89C+0.10	0.18x+0.11	0.25x-0.08
Toxaphene	0.80C+1.74	0.09x+3.20	0.20x+0.22
Aroclor 1016	0.81C+0.50	0.13x+0.15	0.15x+0.45
Aroclor 1221	0.96C+0.65	0.29x-0.76	0.35x-0.62
Aroclor 1232	0.91C+10.79	0.21x-1.93	0.31x+3.50
Aroclor 1242	0.91C+10.79	0.21x-1.93	0.31x+3.50
Aroclor 1248	0.91C+10.79	0.21x-1.93	0.31x+3.50
Aroclor 1254	0.91C+10.79	0.21x-1.93	0.31x+3.50
Aroclor 1260	0.91C+10.79	0.21x-1.93	0.31x+3.50

x' = Expected recovery for one or more measurements of a sample containing concentration C, in µg/L.
s' = Expected single analyst standard deviation of measurements at an average concentration of x, in µg/L.
S' = Expected interlaboratory standard deviation of measurements at an average concentration found of x, in µg/L.
C = True value for the concentation in µg/L.
x = Average recovery found for measurements of samples containing a concentration of C, in µg/L.

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TABLE 5
WEYERHAEUSER DATA FOR PERFORMANCE EVALUATION OF
1 Liter of Water 4/95

Analyte	Amount spike µg/L	standard deviation µg/L on DB1701	standard deviation µg/L on DB608	% recovery DB1701	% recovery DB608
Aldrin	0.2	0.010	0.010	86%	89%
alpha-BHC	0.2	0.009	0.008	78%	81%
beta-BHC	0.2	0.014	0.017	98%	99%
gamma-BHC (Lindane)	0.2	0.010	0.010	77%	78%
delta-BHC	0.2	0.015	0.016	85%	88%
Chlordane	NA	NA	NA	NA	NA
4,4'-DDE	0.2	0.019	0.020	88%	100%
4,4'-DDD	1.0	0.11	0.16	90%	83%
4,4'-DDT	1.0	0.11	0.082	96%	79%
Dieldrin	0.2	0.015	0.015	91%	93%
Endosulfan I	0.6	0.010	0.009	98%	99%
Endosulfan II	0.7	0.065	0.056	94%	88%
Endosulfan sulfate	1.0	0.091	0.085	89%	84%
endrin	1.0	0.089	0.075	96%	83%
Heptachlor	0.2	0.011	0.014	87%	89%
Heptachlor epoxide	0.2	0.012	0.011	87%	88%
Methoxychlor	0.2	0.029	0.027	114%	120%
Toxaphene	NA	NA	NA	NA	NA
PCB-1016	NA	NA	NA	NA	NA
PCB-1221	NA	NA	NA	NA	NA
PCB-1232	NA	NA	NA	NA	NA
PCB-1242	5.0	0.11	0.036	90%	83%
PCB-1248	NA	NA	NA	NA	NA
PCB-1254	NA	NA	NA	NA	NA
PCB-1260	5.0	0.024	0.017	91%	90%

Data based on two sets of four extrations on millipore water
NA = not analyzed

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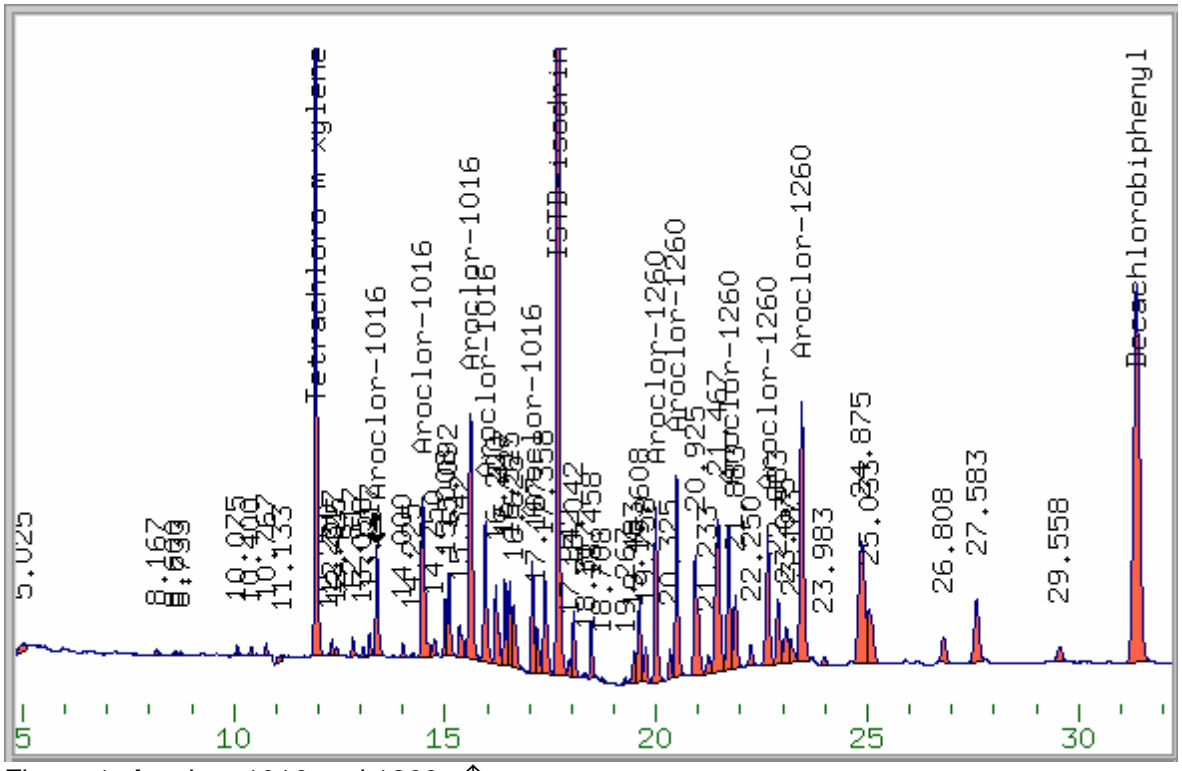

Figure 1: Aroclors 1016 and 1260 ↑

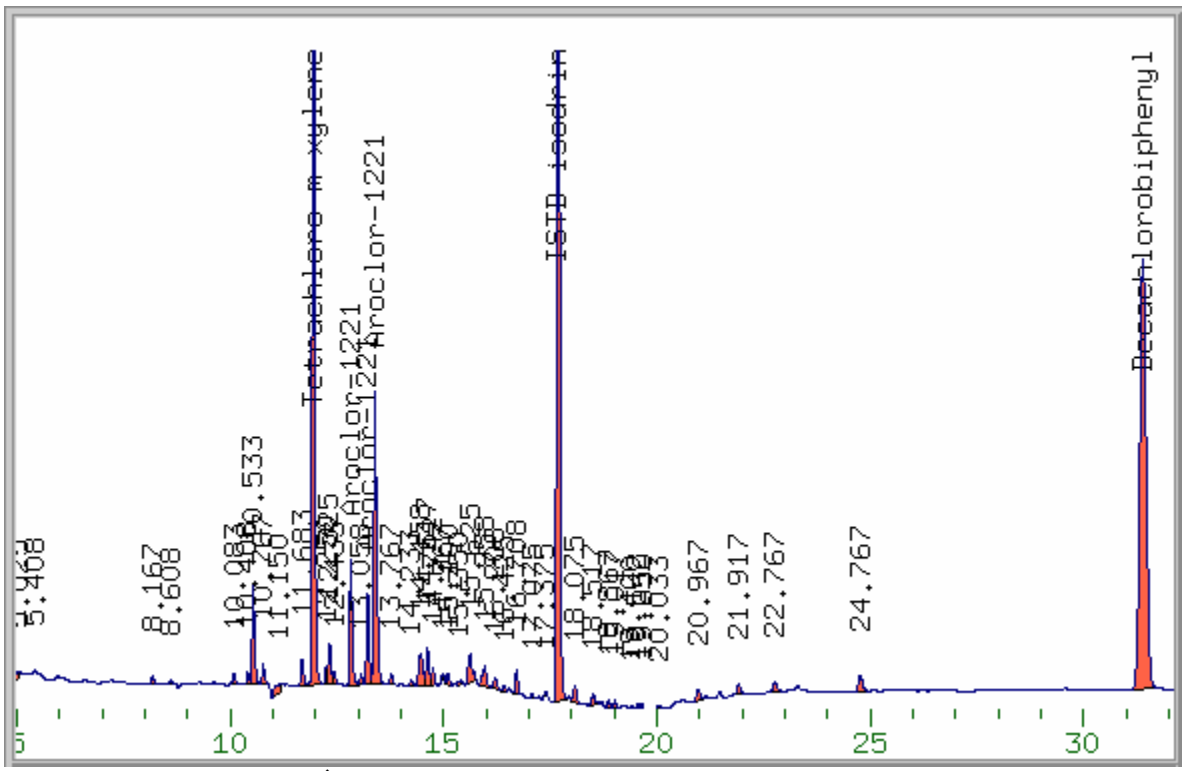

Figure 2: Aroclor 1221 ↑

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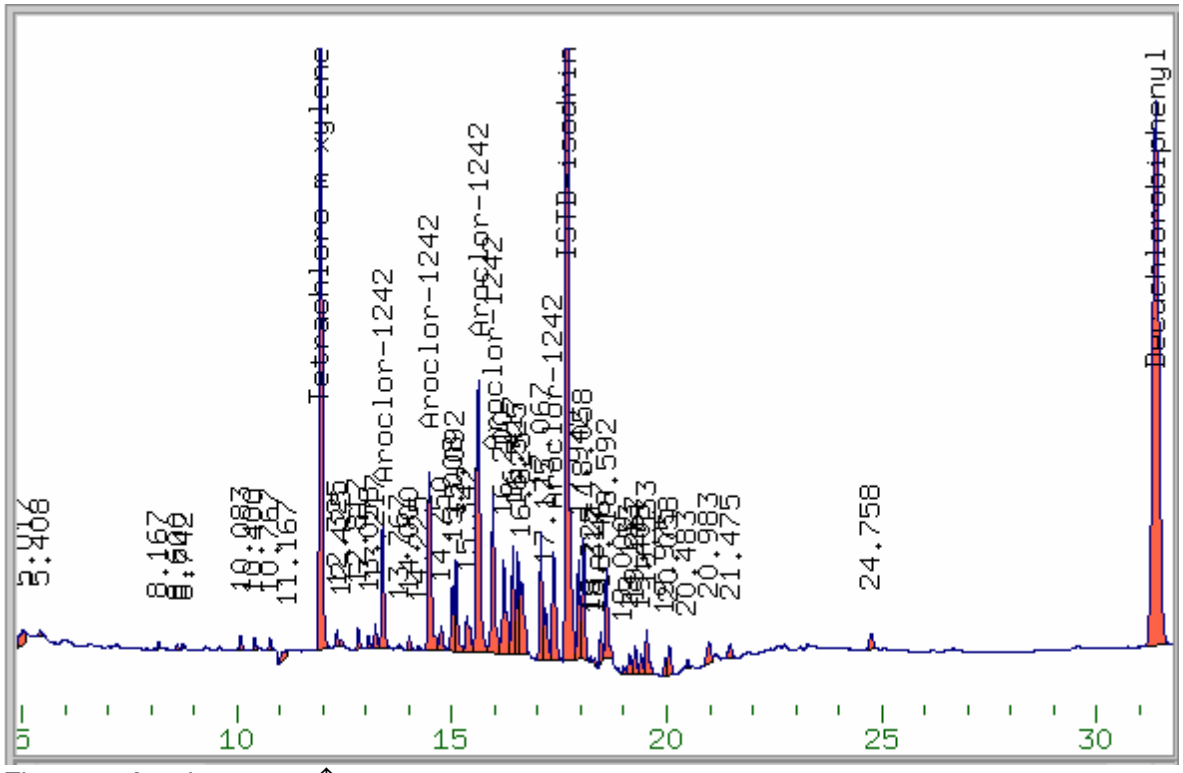

Figure 3: Aroclor 1232 ↑

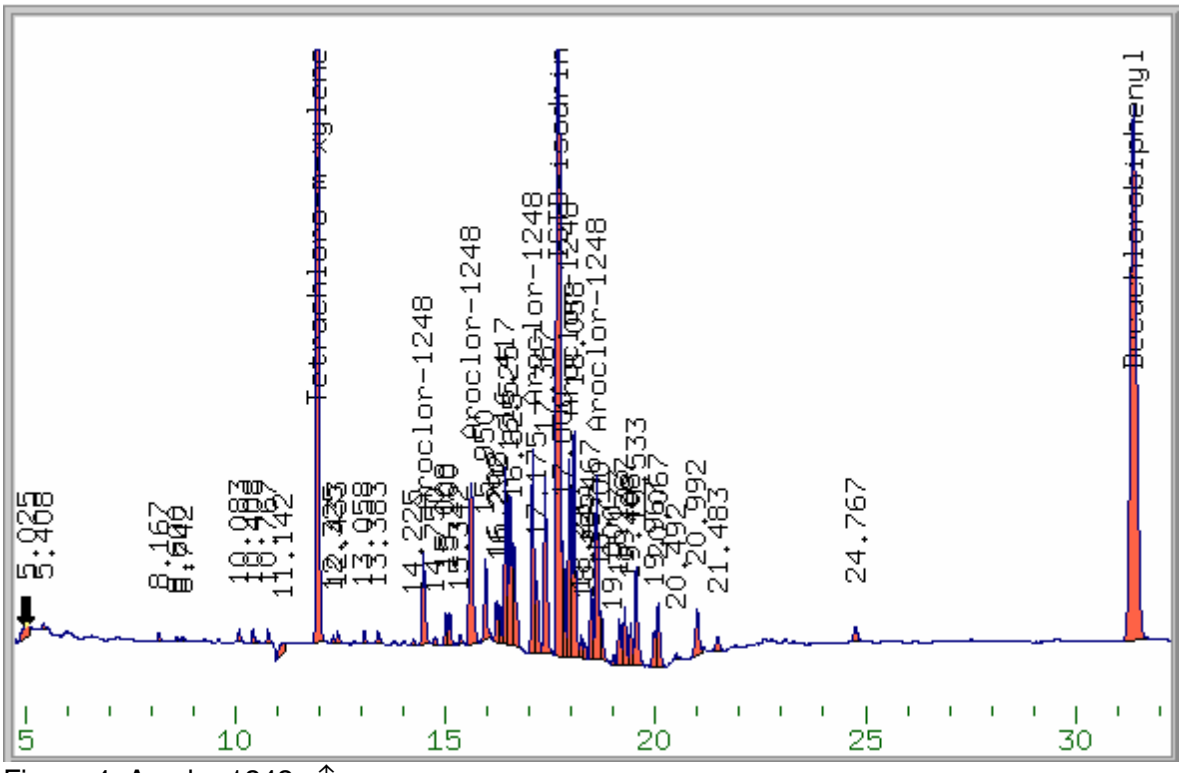

Figure 4: Aroclor 1242 ↑

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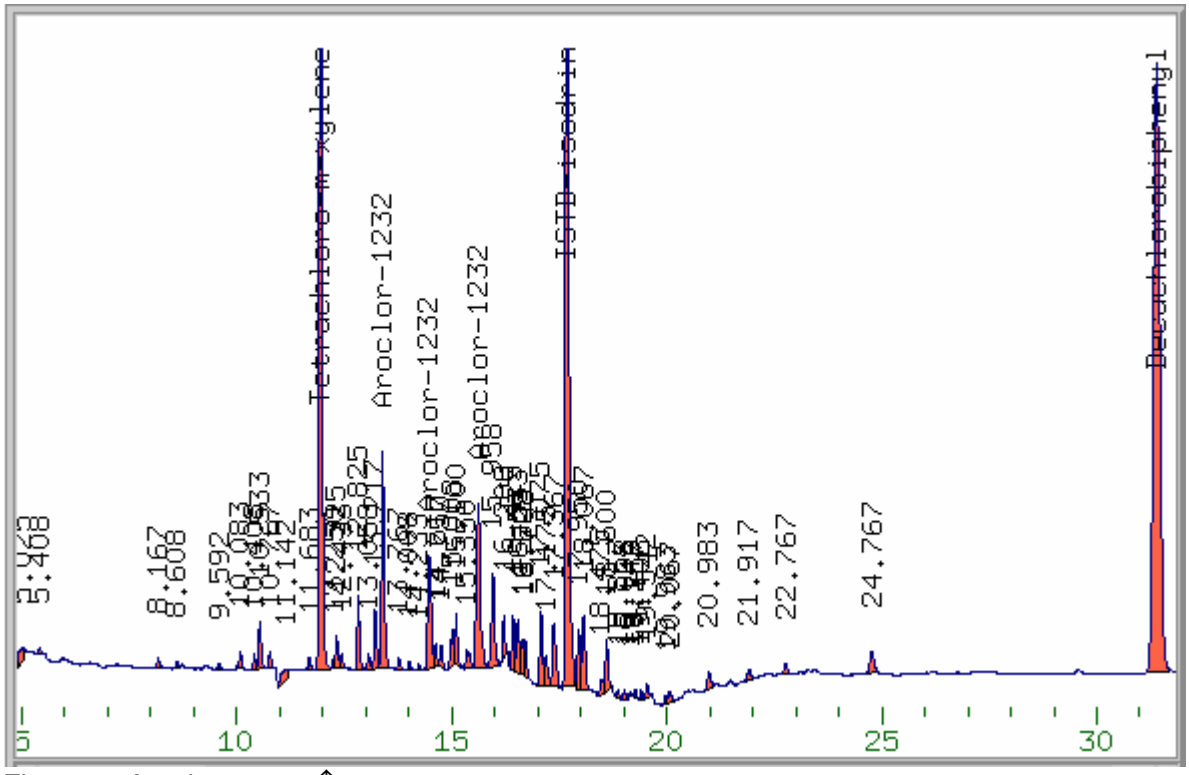

Figure 5: Aroclor 1248 ↑

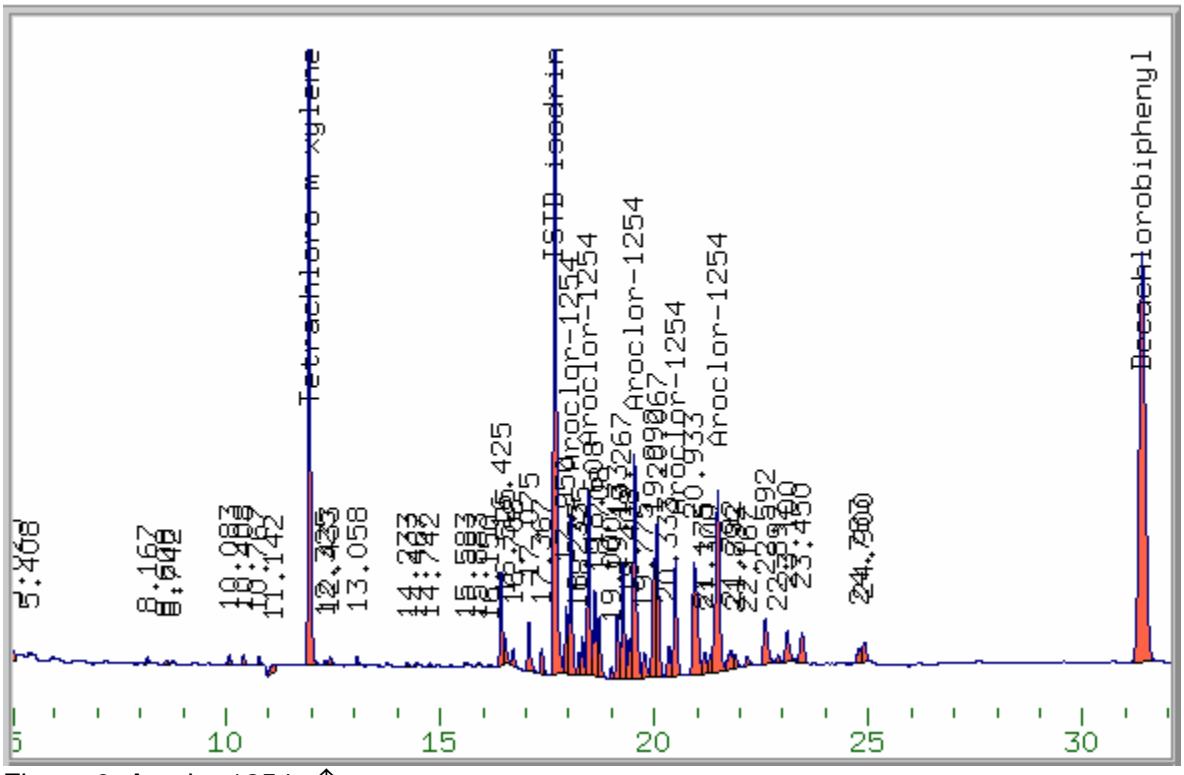

Figure 6: Aroclor 1254 ↑

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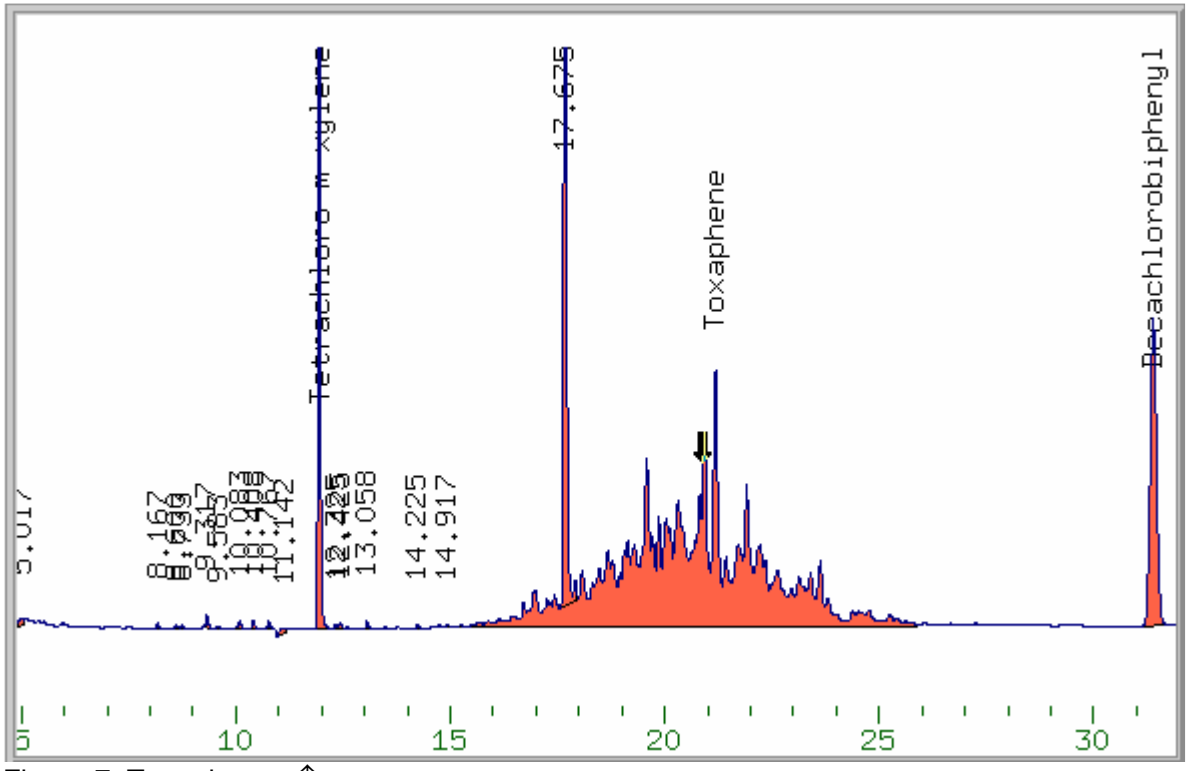

Figure 7: Toxaphene ↑

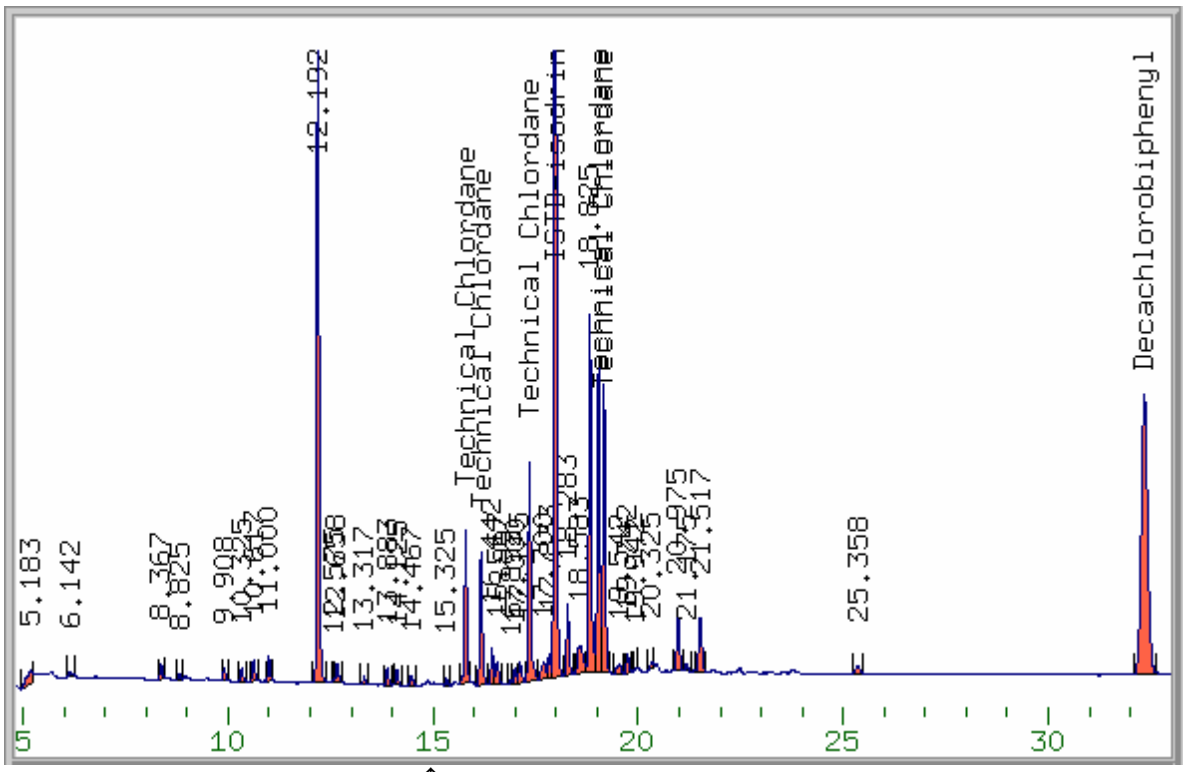

Figure 8: Technical Chlordane ↑

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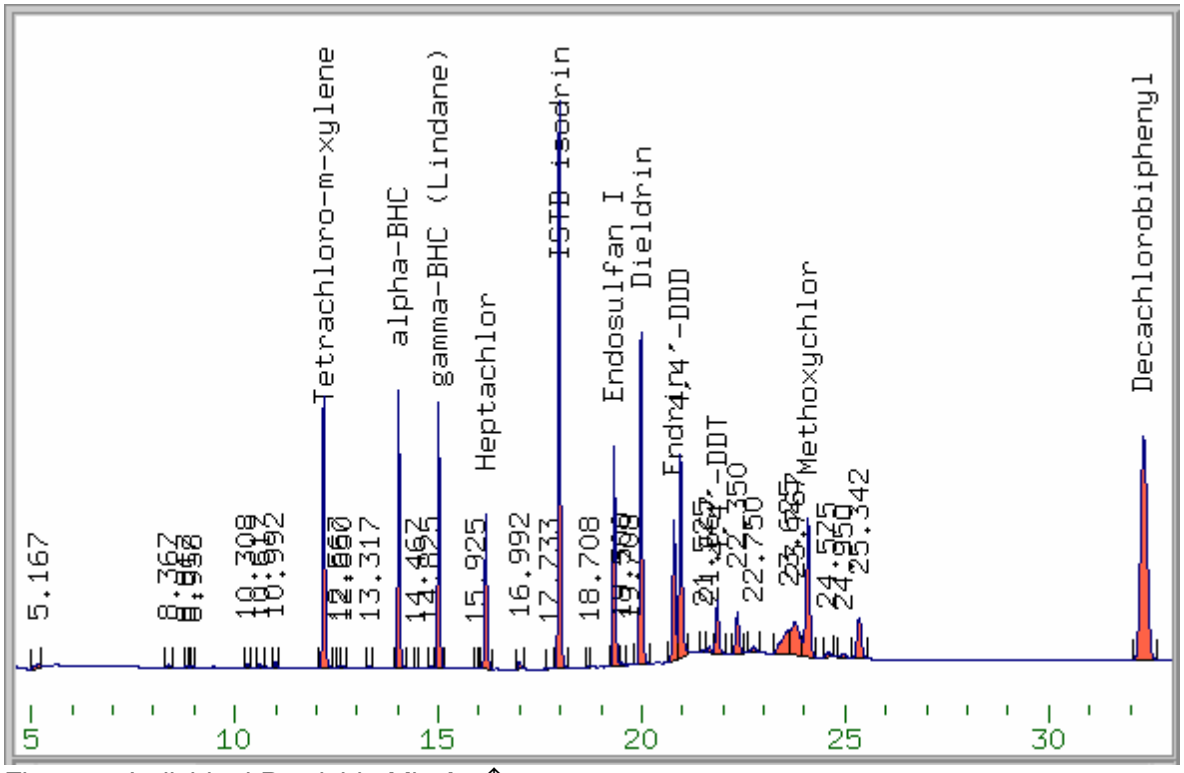

Figure 9: Individual Pesticide Mix A ↑

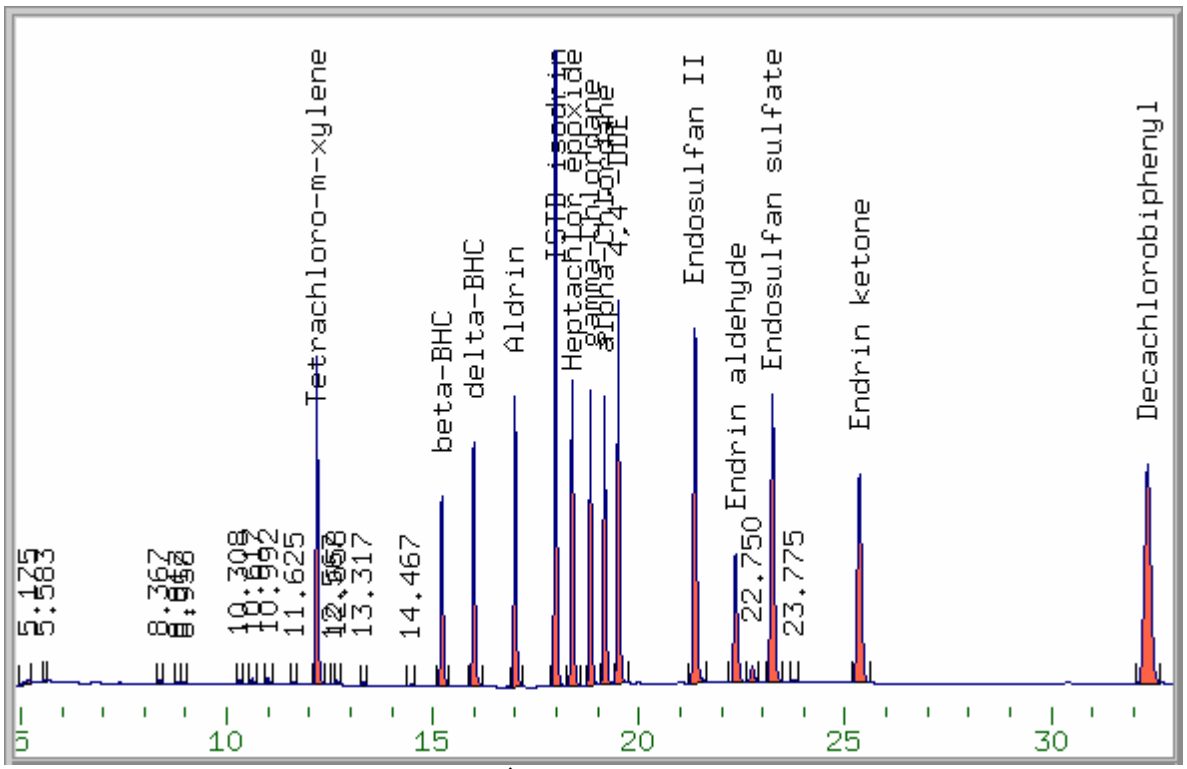

Figure 10: Individual Pesticide Mix B ↑

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W-2

Suspended Solids in Water and Wastewater

Process Owner (TS/PM/OM/LM) **Maxine Ranta**	
Assignment	Reviewed June 18, 2003 by Kathleen Ann LeGreid Reviewed July 13, 2007 by Christine Devine
Electronic only unless colored "Controlled" stamp is present	

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1.0 SCOPE

This method is applicable to the determination of suspended solids in pulp and paper effluents, wood products effluents, raw and treated water and natural waters.

Suspended solids are defined as those solids retained by a standard glass fiber filter.

This method is based upon EPA method 160.2. It replaces method AM1-160.2.

2.0 SUMMARY OF METHOD

A well-mixed sample is filtered through a standard glass fiber filter and the residue retained on the filter is dried to a constant weight at 104 ± 1°C.

3.0 INTERFERENCES

Non homogeneous materials, such as leaves, sticks, and large floating particles, should be excluded from the sample.

Some solids will decompose below the method required temperatures.

Biological organisms, such as algae and insects, are generally not considered to be part of the solids. Problems associated with these should be noted on the report, especially for regulatory monitoring. In some cases, method modifications may be needed to generate an estimate of the suspended solids.

Too much residue on the filter will entrap water and may require prolonged drying times. Clogging of the filter with residue will also prolong filtering time and retain small particles which would normally pass through the filter.

4.0 ESTIMATE OF ANALYTICAL TIME

An average sample will take 10 to 15 min to run. 10 normal samples may be run in 30 to 40 min. Samples with plugging, or high solids problems may take considerably longer, depending upon the nature of the sample.

5.0 SAMPLE SIZE REQUIRED

There is no fixed requirement for sample volume. Normal samples will take about 100 mL. Samples higher in solids may need considerably less. Samples very low in solids may require up to a liter or more; however, greater volumes decrease the accuracy of the test by increasing the amount of materials being washed from the filter. Normally volumes over 500 mL are not recommended.

Include single sample, plus amount needed for QC. This may include a desired amount, in case of need for re-running, or minimum.

6.0 SAMPLING, SAMPLE HANDLING, AND PRESERVATION

Samples should be collected in clean polyethylene or glass containers. Samples should be analyzed as soon as possible after collection, but can be held under refrigeration at < 4°C for seven days, if immediate analysis cannot be performed.

7.0 EQUIPMENT REQUIRED

7.1 Gelman membrane-style filter holder & membrane filter funnel.

7.2 4.7-cm glass fiber filter discs, without organic binder, Gelman type A/E.

7.3 Vacuum source.

7.4 Drying Oven.

7.5 Aluminum pans. (50-mm diameter)

7.6 Graduated cylinders, TC (to-contain) class B

7.7 Desiccator (with Drierite desiccant.)

7.8 Analytical balance capable of weighing to 0.1 mg.

7.9 Vacuum flask, 500-mL or 1-L.

8.0 PROCEDURE

8.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus. Apply vacuum and wash the disc with three successive 20 ± 2-mL volumes of water. This is to remove small loose fibers and wash away wet strength material from the filter. Allow each washing to completely pass through the filter before beginning the next. Continue suction to remove traces of water from filter disc. Release the vacuum. Remove filter disc from apparatus and transfer to a numbered aluminum pan.

8.2 Dry aluminum dish and glass fiber filter for at least one hour at 104 ± 1°C.

WARNING: Skin contact with hot surfaces may burn the skin.

8.3 A supply of filters may be prepared ahead of time and kept in the oven until needed. After drying, handle dish and filter with forceps or tongs only, never the fingers. Place in a desiccator for 20 ± 5 min and then weigh the filter and the pan. Weigh, to the nearest 0.1 mg, immediately before use.

8.4 Assemble membrane filter apparatus with glass filter in place and start suction. Apply a small amount of water to seat filter against the support.

DANGER: Damaged vacuum flasks can implode under high vacuum. Inspect flasks before use for visible signs of cracks, chips, or other signs of abuse. Do not use damaged or injured flasks.

NOTE: Glassware can become "bruised," or weakened by abuse, such as from impact. Discard any vacuum flasks if they have suffered any abuse.

NOTE: Flasks utilizing 'webbing,' tape, or plastic coating is encouraged so that any flying glass would be contained in event of an implosion. (The nature of the liquid being filtered may make this impractical.)

8.5 Shake the sample vigorously and rapidly transfer 100 mL to the graduated cylinder. Record volume to the nearest 1 mL if greater than 20 mL of sample is taken. Pour measured volume through the tared filter.

NOTE: If suspended matter is low, a larger volume may be filtered. With some samples, such as some pulp mill effluents, 100-mL aliquot causes filter plugging and prolonged filtration times. If filtration of 100 mL of sample is not accomplished within a reasonable time (three min), the sample volume should be reduced to allow for filtration within this time frame.

For NPDES, filter smaller increments of the sample and record the time necessary for filtering. Choose the proper sample volume to ensure that filtration is completed just short of the time a significant decrease in filtration rate occurs.

NOTE: For original volumes of less than 10 mL, dilute 10 ± 0.5 mL of the sample to 100 mL or more in volumetric flasks. Run the test on the diluted sample.

8.6 Rinse the graduated cylinder and filter holder three times with 10 ± 2-mL volumes of water. Allow the washing to completely pass through the filter before beginning the next.

8.7 Release the vacuum.

8.8 Remove the filter from the membrane filter assembly, being careful not to lose any of the sample, and place in the tared aluminum pan. Discard filtrate down the sink.

8.9 Dry 4 to 24 hr at 104 ± 1°C. Cool in a desiccator for 20 ± 5 min and weigh the pan and filter.

WARNING: Skin contact with hot surfaces may burn the skin.

8.10 Samples are dried overnight and historical data has shown that this is sufficient to reach a constant weight. If a shorter drying time is used or at client request, repeat step 8.9 to make sure there is less than 0.5 mg change of mass in the filter. Record the service request number for this information in the Laboratory Standard Recovery notebook.

8.11 Run one blank, a control, and one sample in duplicate in conformance with laboratory guidelines, with a minimum of one of each of these per batch or every 10 samples, whichever is more frequent. The blank is done without running any water through the filter except for the initial rinse.

The blank generally has a loss of 0.1 mg, which is <u>not</u> subtracted from the samples. If the blank gains mass or loses more than 0.2 mg on a 47 mm filter, the samples should be redone.

The controls shall meet the established QC requirements for that control sample. The manufacturer supplies acceptance limits with each lot of standard. Duplicates shall meet the QC requirements for that type of sample.

NOTE: The current control is NSI Standard Reference Material. The recovery limits are 85-115%.

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9.0 QUALITY ASSURANCE

Quality assurance is addressed in part 8.11 above.

10.0 REPORT

10.1 Calculations:

Calculate suspended solids as follows:

$$\text{Suspended solids, mg/L} = \frac{(A - B)(1{,}000{,}000)}{C}$$

Where:

A = Mass of filter/pan and residue in g.
B = Mass of filter/pan in g.
C = Volume of sample filtered in mL.

10.2 Do not report more than 3 significant figures.

10.3 Results should be reported in mg/L.

10.4 The lower detectable amount of material is dependent upon the amount of the initial starting volume. The mass of residue used to calculate the detection limit is 1mg.

Example: (0.0010g) (1,000,000) ÷ 500mL = 2 mg/L
2 mg/L would be the detection limit for a 500-mL aliquot.

10.5 Precision and Accuracy

There is no way to determine the precision and accuracy for this method since it generates a method defined value. There is also no defined standard with which to make an objective determination of TSS recovery.

The precision of the determination varies directly with the concentration of suspended solids and degree of difficulty in obtaining a representative sample for analysis. Analysis of typical Weyerhaeuser wood products and pulp mill waste streams by WATS Laboratory showed a standard deviation of ± 1 mg/L (14 % - 11 replications) at 7 mg/L, ± 2 mg/L (4 % - 6 replicates) at 52 mg/L, and ± 3 mg/L (3% - 12 replications) at 100 mg/L.

The results of twenty 94.2 mg/L TSS standard run over a period of 7 months (Oct. 2006-April 2007) gave a recovery of 92.2 mg/L with a standard deviation of 2.1 mg/L.

11.0 KEY WORDS

glass fiber, filter, non-filterable solids, solids, suspended solids, total suspended solids, TSS, water, wastewater

12.0 REFERENCES

12.1 Standard Methods for the Examination of Water and Wastewater, 18th ed., p. 2-56, Method 2540D.

12.2 EPA Manual of Methods for Chemical Analysis of Water and Wastes, March 1983, p. 160.2.

12.3 "Precision of the TSS Test," Weyerhaeuser Technical Report by S. Vincent, February 1977.

12.4 ASTM method D5907.

13.0 REVISION HISTORY

13.1 7/13/07 8.9 – changed the drying time; 8.10 - changed wording to eliminate requirement to check constant weight; 8.11 – added information about QC requirements; 10.5 – Updated precision and accuracy

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W-3

Total and Total Soluble Phosphorus in Water

Process Owner (TS/PM/OM/LM) **Maxine Ranta**	
Assignment	Reviewed by Christine Devine September 9, 2004 Reviewed by Christine Devine August 19, 2008
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1.0 SCOPE

1.1 This method is applicable for the analysis of total and total soluble phosphorus in pulp and paper effluents, wood products effluents, raw and treated waters, and natural waters. Total phosphorus includes all of phosphorus present in the sample regardless of form, as measured by the persulfate digestion procedure.

1.2 This method is capable of measuring concentrations of phosphorus from 0.01 to 0.75 mg P/L. This range can be extended by diluting the sample.

1.3 This method replaces AM 1-365.3.

2.0 SUMMARY OF METHOD

Polyphosphates and organic phosphates are converted to orthophosphates by sulfuric acid/persulfate digestion. Ammonium molybdate and antimony potassium tartrate react 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 heteropoly complex by ascorbic acid. The color is proportional to the phosphorus concentration present in the standard or sample.

NOTE: The heteropoly is not a true dissolved color, but is a fine precipitate.

3.0 INTERFERENCES

3.1 Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. This can be removed by the addition of potassium iodide during the digestion stage.

3.2 Hexavalent chromium and nitrate interfere to give results about 3 % low at concentrations of 1.0 mg/L and 10-15 % low at concentrations of 10 mg/L.

3.3 Sulfide and silicate do not interfere in concentrations of 1.0 and 10.0 mg/L, respectively.

4.0 ESTIMATE OF ANALYTICAL TIME

For a single sample the time estimate is 1.5 hours. The best batch size is 15 samples and the time estimate is 6 hours for the digestion, color development, and calculation.

5.0 SAMPLE SIZE REQUIREMENT

250 mL of sample volume collected in a plastic bottle is needed to meet analysis and quality control requirements.

6.0 SAMPLING, SAMPLE HANDLING AND PRESERVATION

If immediate analysis is not possible, preserve the samples by acidifying to pH < 2 with 5 mL of 9 *N* H_2SO_4 per liter. Store at 4 °C. Samples for regulatory use may be held in this manner for no longer than 28 days from sample collection.

7.0 EQUIPMENT REQUIRED

CAUTION: All glassware should be new and not have been used for other analyses. It should be washed separately from other glassware to avoid contamination. Care must be taken to avoid the use of phosphate soaps in the laboratory. See AP O-GlassClean.

7.1 Spectrophotometer with 1-cm and 5-cm quartz cells for measurements at 650 nm.

7.2 Filtration and centrifugation apparatus.

7.3 125-mL Phillips beaker.

7.4 Millipore filtration apparatus.

7.5 0.45-µm Millpore filter disc - 47-mm diameter.

7.6 Glass scoop that will deliver approximately 0.4 g.

8.0 REAGENTS REQUIRED

8.1 Ammonium molybdate-antimony potassium tartrate solution: In a 250-mL volumetric flask dissolve 2.0 ± 0.1 g of $(NH_4)_6\ Mo_7O_{24}{\bullet}4H_2O$ and 0.05 ± 0.005 g of $K(SbO)C_4H_4O_6.1/2\ H_2O$ in 100 - 200 mL of water. Dilute to volume with water. Mix well. This solution is stable for 6 months.

See WARNING under ammonium molybdate in the appendix.

See WARNING under antimony potassium tartrate in the appendix.

8.2 Ascorbic acid solution: In a 100-mL volumetric flask dissolve 6 ± 0.1 g ascorbic acid in 75 - 90 mL of water. Dilute to volume with water. Mix well. Make fresh daily.

See WARNING under ascorbic acid in the appendix.

8.3 Sulfuric acid, 11 *N*: In a 1000-mL volumetric flask slowly add 310 ± 1mL of conc H_2SO_4 to 550 - 650 mL of water while stirring constantly. Cool and dilute to 1000 mL. Mix well.

WARNING: SOLUTION WILL BECOME VERY HOT. THE FLASK SHOULD BE PLACED IN AN ICE BATH (WITHIN A HOOD) AND STIRRED CONSTANTLY. PROTECTIVE CLOTHING SUCH AS GLOVES, APRON, AND FACE SHIELD SHOULD BE WORN.

See DANGER under sulfuric acid in the appendix.

8.4 Sulfuric acid dilution solution: In a 500-mL volumetric flask add 10 ± 0.1mL of 11 *N* H_2SO_4 to approximately 350 - 450 mL of water. Dilute to volume with water. Mix well.

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See DANGER under sulfuric acid in the appendix.

8.5 Ammonium Persulfate.

See WARNING under ammonium persulfate in the appendix.

8.6 Phosphorus stock solution, 50 mg P/L : In a 1000-mL volumetric flask dissolve 0.2195 g of pre-dried KH_2PO_4 (anhydrous) (105 ± 2 °C for 1hour to overnight) in 800 – 900 mL of water. Dilute to volume with water. Mix well. This solution is stable for 6 months. 1 mL of this solution = 50 µg P.

See WARNING under potassium phosphate (monobasic) in the appendix.

8.7 Phosphorus standard solution, 2.5 mg P/L: Prepare fresh daily by pipeting 5 mL of phosphorus stock solution (8.6) into a 100 mL volumetric flask and dilute to volume with water. Mix well. 1 mL of this solution = 2.5 µg P.

See WARNING under potassium phosphate (monobasic) in the appendix.

9.0 PROCEDURE

9.1 Digestion

9.1.1 Transfer a maximum of 50 mL of sample, or a smaller aliquot, into a Phillips beaker and adjust the pH of the sample using a pH electrode to between pH 5 to 8.

9.1.2 If total soluble phosphorus is to be performed, filter the sample through a 45µm filter and transfer a maximum of 50 mL of sample, or a smaller aliquot, into a Phillips beaker and adjust the pH of the sample using a pH electrode to between pH 5 to 8.

9.1.3 If the sample is to be taken back to a final volume of 50 mL, add 1 mL of the 11 *N* H_2SO_4 to the flask and mix well. If the sample is to be taken back to a final volume of 25 mL, add 0.5 mL of the 11 N H_2SO_4 and mix well.

CAUTION: Improper acidity will retard or enhance color formation.

9.1.4 Add 0.4 g of ammonium persulfate (one level scoop) to the flask, mix and place on a hot plate to boil gently for approximately 30-40 min or until a final volume of 10 - 12 mL is reached. Alternately the samples can be heated in an autoclave at 121 ± 2°C at 100-140 kPa (15-20 psi).

9.1.5 Filter samples through a 0.45µm filter and collect the filtrate in a 50-mL graduated test tube. Rinse the beaker and filter with several aliquots of water. Bring the sample to a final volume of 25 or 50 mL. Pour the sample back into the Phillips beaker and mix well. Rinse the tube and continue with the next sample.

9.1.6 Blanks, controls, and standards for the calibration curve must be carried through the digestion procedure.

9.1.7 The high level calibration curve should be prepared by diluting the following aliquots to a final volume of 50 mL using the same 50-mL graduated test tube used for the samples.

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mL 2.5 mg/L Standard Solution	Standard Concentration (mg/L)
15	0.75
10	0.50
5.0	0.25
2.0	0.10
1.0	0.050
0	0

9.1.8 The low level calibration curve should be prepared by diluting the following aliquots to a final volume of 50 mL using the same 50-mL graduated test tube used for the samples.

mL 2.5 mg/L Standard Solution	Standard Concentration (mg/L)
5.0	0.25
2.0	0.10
1.0	0.050
0.50	0.025
0.20	0.010
0	0

9.1.9 Spikes and duplicates are run on every 10th sample, or one per batch or service request, whichever is more frequent. ICV and ICB standards are run immediately after the calibration curve with CCV and CCB standards run after every 10 samples and at the end of the run.

9.2 Manual Spectrophotometric Determination

9.2.1 Pipet 10 mL of the digested sample and standards, or an aliquot to be diluted to 10 mL, into a 15-mL disposable test tube. Dilutions must be performed using the sulfuric acid dilution solution in section 8.4.

9.2.2 Add 0.8 mL of ammonium molybdate-antimony potassium tartrate solution, cap and mix.

9.2.3 Add 0.4 mL of ascorbic acid solution, cap and mix.

9.2.4 After 5 min, measure absorbance at 650 nm in a 1-cm cell with a spectrophotometer and determine the phosphorus concentration from the standard curve. The color is stable for 45 minutes to one hour. Concentrations in the range of 0.10 to 0.75 mg/L may be determined using the 1-cm cell. The linear calibration curve should have a correlation coefficient (r) of at least 0.995.

NOTE: Do not filter sample after color formation. The heteropoly is not a true dissolved color, but is a fine precipitate.

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9.2.5 If samples are known to contain lower amounts of phosphorus, < 0.1 mg/L, pipet 25 mL of the digested sample into a 30-mL disposable beaker containing a stir bar.

9.2.6 Add 2 mL of ammonium molybdate-antimony potassium tartrate solution and mix.

9.2.7 Add 1 mL of ascorbic acid and mix.

9.2.8 Repeat step 9.2.4 above using a 5-cm cell. Concentrations in the range of 0.01 to 0.25 mg/L should be determined using the 5-cm cell. The linear calibration curve should have a correlation coefficient (r) of at least 0.995.

10.0 QUALITY CONTROL

Normally expected QC parameters are as follows:

10.1	Calibration curve – Coefficient of Correlation (r)	0.995
10.2	ICV and CCV recovery	100 ± 10%
10.3	ICB and CCB	< 0.01
10.4	Spike Recovery	100 ± 25%
10.5	Duplicate RPD	20%

11.0 REPORT

11.1 CALCULATION:

$$\text{P, mg/L} = \frac{(\text{mg/L P from curve})(\text{Final volume, mL})}{(\text{mL sample})}$$

11.2 Report the final value to two decimal places with no more than three significant figures. Values near the detection level should only have one significant figure.

11.3 Report results as total phosphorus (TP) or total soluble phosphorus (TSP) in mg/L.

11.4 PRECISION AND ACCURACY

From the most recent 15 runs, expected precision for the high level curve is 2.0 % RSD at a 10X dilution of the 3.50 mg/L standard and an expected accuracy of 95.1 ± 1.9 %.

From the most recent 14 runs, expected precision for the low level curve is 1.8 % RSD at a 50X dilution of the 3.50 mg/L standard and an expected accuracy of 95.3 ± 1.7%.

12.0 KEY WORDS

AM 1-365.3, AM E-365.3 TP, phosphorus, total soluble phosphorus

13.0 REFERENCES

13.1 Methods for "Chemical Analysis of Water and Wastes," revised March 1983.

13.2 "Standard Methods for the Examination of Water and Waste Water," 18th Edition, 1992.

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APPENDIX

Ascorbic acid

WARNING: Ascorbic acid is relatively non-hazardous in routine laboratory situations. It is not expected to present significant health risks to the workers who use it.
Avoid inhalation: May cause mild irritation to the respiratory tract.
Avoid eye and skin contact: May cause mild irritation.
Fire is possible at elevated temperatures or by contact with an ignition source.
OSHA Permissible Exposure Limit (PEL): 15 mg/m^3 total dust, 5 mg/m^3 respirable fraction for nuisance dusts
Aqueous solutions are rapidly oxidized by air.
Incompatibilities: Strong oxidizers and alkali hydroxides, alkalis, iron, copper, sodium salicylate, sodium nitrite, theobromine, and methenamine.

Ammonium molybdate

WARNING: Avoid inhalation: Ammonium molybdate is an irritant to mucous membranes and the upper respiratory system. Pungent taste in mouth and throat, coughing, labored breathing. Can be a route of absorption by the body with possible symptoms of sore throat, abdominal pain, nausea. May cause anemia, gout, headaches, weight loss, joint pain, and liver or kidney damage.
Avoid skin contact: Causes irritation to skin. Symptoms include redness, itching, and pain.
Avoid eye contact: causes irritation, redness, and pain.
It appears the chemical, physical, and toxicological properties have not been thoroughly investigated.
OSHA permissible exposure limit (PEL): 5 mg/m^3 for soluble molybdenum compounds as Mo, 15 mg/m^3 for insoluble molybdenum compounds as Mo.
Hazardous decomposition products: burning may produce ammonia, nitrogen oxides, and Mo metal fumes.
Incompatible with: alkali metals, strong oxidizing agents, and strong acids.

Ammonium persulfate

WARNING: Avoid breathing dust. Ammonium persulfate can cause irritation to the mucous membranes and respiratory tract. Symptoms may include sore throat, coughing, shortness of breath. Repeated or prolonged exposure may cause asthma.
Avoid skin contact. May cause irritation to skin. Symptoms include redness, burning sensation, and pain. May cause dermatitis, burns, and moderate skin necrosis. Persulfate allergy is not uncommon and manifest itself in the form of a skin rash.
Eye Contact can cause irritation, redness, and pain. May cause burns.
Ingestion can cause nausea, diarrhea, vomiting and sore throat.
Substance is a strong oxidizer and its heat of reaction with reducing agents or combustibles may cause ignition. Contact with oxidizable substances may cause extremely violent combustion. Also may act as initiation source for dust or vapor explosions.
Incompatable with powdered metals, phosphorus, hydrides, organic matter, halogens, acids, strong alkalis, strong reducing agents, alcohols, organics, or other readily oxidizable materials.
Avoid heat and moisture.
Unstable. Gradually decomposes losing oxygen. Decomposes more rapidly at higher temperatures producing toxic and corrosive fumes including ammonia, nitrogen oxides and sulfur oxides. If in solution reacts violently with iron, powdered alumiinium and silver salts. The solution in water is a medium strong acid. Metals other than stainless steel are apt to cause decomposition of persulfate solutions.

Antimony potassium tartrate

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WARNING: Avoid inhalation of antimony potassium tartrate dust. Toxic if inhaled. Symptoms of exposure: May cough, nausea, diraahea, metallic taste, salivation, skin rash, severe liver damage, death.
Avoid skin contact: May cause skin irritation.
Avoid eye contact: May cause eye irratation. May cause liver damage.
Avoid contact with acids (may yield SbH_3) and oxidizers (may be explosive with oxides of nitrogen and chlorine, and permanganate.)

Potassium phosphate (monobasic)

WARNING: Avoid inhalation off potassium phosphate dust: May cause mild irritation to the respiratory tract.
Avoid eye contact: May cause irritation, redness, and pain.
Avoid skin contact: Irritant due to its acidic nature. May cause inflammation and pain on prolonged contact, especially with moist skin.
Chronic Exposure from absorbance may sequester calcium and cause calcium phosphate deposits in the kidneys, however this is unlikely since phosphates are slowly and incompletely absorbed (when ingested), and seldom result in systemic effects. The toxicity of phosphates is because of their ability to sequester calcium.
OSHA Permissible Exposure Limit (PEL): 15 mg/m^3 total dust, 5 mg/m^3 respirable fraction for nuisance dusts.
Incompatible with moisture and strong oxidizing agents.

Sulfuric acid

DANGER: Degree of hazard depends upon concentration. Conc. acid mainly refers to those over 52 %. Handle with care. Wear safety goggles and a plastic apron. Use in a hood if mist or fumes generated. Conc. sulfuric acid is a strong, corrosive acid and can cause severe burns. Very carefully add acid to water with frequent or continuous stirring. Mix in a tub or stoppered sink. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.
Conc. sulfuric acid is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes and skin.
Inhalation of mist or heated fumes may result in spasm, inflammation and edema of the larynx and bronchi, chemical pneumonitis and pulmonary edema. Symptoms of exposure may include burning sensation or irritation of the nose and throat, coughing, wheezing, laryngitis, shortness of breath labored breathing, headache, nausea and vomiting.
Avoid skin contact. Conc. acid is extremely corrosive. Symptoms of redness, pain, and severe burn can occur. Circulatory collapse with clammy skin, weak and rapid pulse, shallow respirations, and scanty urine may follow skin contact or ingestion. Circulatory shock is often the immediate cause of death.
Avoid eye contact. Extremely corrosive. Sulfuric acid can cause severe irritation or burns and result in permanent damage. Contact can cause blurred vision, redness, pain and severe tissue burns. Strong concentrations will probably cause blindness.
Long-term exposure may cause erosion of teeth.
Sulfuric acid is an oxidizer and strong dehydrating agent. May cause ignition of finely divided organic materials on contact. Reacts with most metals to produce hydrogen gas, which can form an explosive mixture with air. Keep away from bases, carbides, chlorates, fulminates, nitrates, picrates, cyanides, alkali halides, zinc iodide, permanganates, hydrogen peroxide, azides, perchlorates, nitromethane, phosphorous, nitrites, halogens, metal acetylides, oxides and hydrides, metals (especially finely powdered ones) (yields hydrogen gas), strong oxidizing and reducing agents, water, and many other reactive substances.
Harmful if absorbed through skin.
Reaction products include hydrogen, from reaction with metals, or sulfur oxide fumes when heated.
Permissible exposure limit (PEL) is 1 mg/m^3.
Mist is a known carcinogen, depending on duration and level of exposure.

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W-4

Polychlorinated Biphenyls Analysis by GC/ECD

Process Owner (TS/PM/OM/LM) **Randy Eatherton**	
Assignment	Reviewed June 20, 2003 by Christine Devine Reviewed December 4, 2007 by Christine Devine
Electronic only unless colored "Controlled" stamp is present	

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1.0 SCOPE

1.1 Method 8082 (based on EPA's SW-846 method 8082, revision 0, December 1996) is used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors ® in extracts from solid and liquid matrices. Analysis is employed with dual Megabore capillary columns in conjunction with electron capture detectors (ECD).

CAS #	ANALYTE
12674-11-2	Aroclor-1016
11104-28-2	Aroclor-1221
11141-16-5	Aroclor-1232
53469-21-9	Aroclor-1242
12672-29-6	Aroclor-1248
11097-69-1	Aroclor-1254
11096-82-5	Aroclor-1260

1.2 Table 1: Weyerhaeuser method detection limits (MDL) for Aroclor 1242, in water and soil/sediment matrices, for the wide-bore capillary column version of this method. The MDLs for the Aroclor analysis of a specific sample may differ from those listed in Tables 1 because they are dependent upon the nature of interferences in the sample matrix.

1.3 Aroclor identification is based on single injector, dual column/dual detector analysis. Quantitation is done on both columns providing qualitative and quantitative confirmation. (If the relative percent deviation is greater than 25 percent, the results are flagged.)

1.4 Aroclors are multi-component mixtures and require pattern recognition for identification. 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") which alters the peak patterns.

2.0 SUMMARY OF METHOD

2.1 A measured volume or mass of sample is extracted using the appropriate sample extraction technique specified in Methods AC G-3510, AC G-3540, AC G-3550, and AC G-3580. The amount used is approximately 0.5 L for liquids, 2 g to 30 g for soils, 30 g to 150 g for sediments, 10 grams for pulp/paper and 0.1 gram for oil. All samples are extracted with methylene chloride. Liquid samples are extracted using a separatory funnel (AC G-3510); soil samples are extracted using ultrasonic extraction (AC G-3550); and pulp/paper samples are extracted with soxhlet extraction (AC G-3540). For all soil/sediment and tissue samples, GPC (gel permeation

chromatography AC G-3640) is optional but sulfuric acid clean up is mandatory. For all solid extracts, Florisil clean up (AC G-3620) is mandatory. After clean-up, the extract is analyzed by injecting a 1- to 2-µL sample into a gas chromatography with dual wide-bore fused silica capillary columns and dual electron capture detectors (GC/ECD).

2.2 The MDLs - achievable in routine analyses of complex samples using this method - will usually be dependent on the degree of interference associated with the presence of co-eluting compounds to which the ECD will respond. This interference will have more influence than on the inherent limitations in detector performance, or the irreducible noise associated with instrument electronics.

If interferences prevent identification and qualification of the analytes within quality control (QC) limits at relevant concentrations, the samples must have additional cleanup and/or be diluted to determine a true quantitation limit based on the interferences of the Aroclor patterns.

3.0 INTERFERENCES

3.1 Refer to Methods AC G-3550, AC G-3600, and AI G-8000.

3.2 Sources of interference in this method can be grouped into three broad categories: contaminated solvents, reagents, or sample processing hardware; contaminated GC carrier gas, parts, column or detector surfaces; and the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Knowledge of good laboratory practices is assumed, including steps to be followed in routine testing and clean up of solvents, reagents and sample processing hardware, and instrument maintenance. The discussion that follows focuses on sources of interference associated with the sample matrix and compound classes that represent common sources of interference, particularly phthalate esters, organosulfur compounds, lipids, and waxes. Interferences co-extracted from the samples will vary considerably. While general clean-up techniques are referenced or provided as part of this method, unique samples may require additional clean-up approaches to achieve desired degree of discrimination and quantitation.

3.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. These materials may be removed prior to analysis using Gel Permeation Clean up - pesticide option (AC G-3640). Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interferences from phthalate esters. Exhaustive clean up of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

3.4 The presence of elemental sulfur will result in large peaks that interfere with the detection of later eluting Aroclors. AC G-3660 is used for removal of sulfur, where activated copper is employed to remove sulfur.

Sulfur cleanup is mandatory with sediment samples due to the high sulfur content.

3.5 Wax, lipids other high molecular weight co-extractables can be removed by sulfuric acid clean up (AC G-3665) and by Gel-Permeation clean up (AC G-3640).

3.6 Pulp and paper samples extracted with methylene chloride result in small fibers being suspended, which must be removed with sulfuric acid clean up (AC G-3665).

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3.7 All soil, sediment, oil and pulp/paper Aroclor extracts are subjected to Florisil cartridge cleanup (AC G-3620) to remove polar compounds.

3.8 Cross-contamination of glassware. High levels of Aroclors can easily contaminate low-level samples. Care must be exercised when handling samples that came from transformers or around transformers as these can contain % levels of Aroclors. If such samples are being analyzed, do not use Florisil clean up (contamination of the manifold will happen). Dilute the sample by 10,000 for analysis. All glassware that was used must be rinsed several times into a hazardous waste container. The glassware must be clean before it is sent to glass washing.

CAUTION: Do not oven dry glassware. Drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized in the oven and can spread to other glassware.

4.0 ESTIMATE OF ANALYTICAL TIME

4.1 Oil samples require only dilution, so the time is less than ½ hour for a batch of 12. Pulp/paper samples require soxhlet extraction and multiple cleanups, requiring 4 man-hours per batch, 30 hours elapsed time per batch of 12. Water samples are dependent on emulsions formed during extraction. No emulsions require 2 hours prep time per batch of 6. Emulsions require 4 hours prep time per batch of 6. Soil samples with no fines would require 2 hours prep time per batch of 12. Samples containing fines, resulting in plugged up filter funnels, could add hours to the prep time.

5.0 AMOUNT OF SAMPLE REQUIRED

Soil samples require 200 grams; sediment requires 500 grams; water requires 2 liters; oil requires 2 grams and pulp/paper samples require 30 grams per sample. This allows enough sample for matrix spike/matrix spike duplicate.

6.0 SAMPLING, SAMPLE HANDLING, AND PRESERVATION

6.1. Water samples must be extracted within 7 days from date of sampling. Soil/sediment samples must be extracted within 14 days from date of sampling. Water and soil/sediment samples are stored under refrigeration (4 ±2°C). Tissues, pulp, paper and oil samples have no defined holding time.

6.2 Extracts must be stored under refrigeration (4 ±2°C) and analyzed within 40 days of extraction.

7.0 EQUIPMENT REQUIRED

7.1 Glassware (see Methods AC G-3510, AC G -3540, AC G -3550, AC G -3620, AC G -3640, and AC G -3660 for specifications).

7.2 Kuderna-Danish (K-D) or TurboVap© apparatus for concentration of initial extracts.

7.3 Gas chromatography analytical system complete with gas chromatography (Agilent 5890) with split/splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detector, and data system (Chemstation and Target). The columns are equipped with 5 meters of deactivated 0.54-mm fused silica guard columns.

7.3.1 Wide-bore columns.

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7.3.2 Column 1 - 30 m x 0.53-mm ID fused silica capillary column chemically bonded (Rtx-CLPesticides) 0.50-µm film thickness.

7.3.3 Column 2 - 30 m x 0.53-mm ID fused silica capillary column chemically bonded (Rtx-CLPesticides 2) 0.42-µm film thickness.

7.3.4 Wide-bore columns are installed in 6.35-mm (1/4-inch) injectors with Siltek Drilled Uniliner (Restek #21055-214.5) deactivated liners designed specifically for use with these columns. The columns are connected to the 5-m guard column with a press fit Y-shaped 3-way fused silica union.

7.3.5 Alternate Column 1 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 35 % phenyl methylpolysiloxane (DB 608), 0.83-µm film thickness.

7.3.6 Alternate Column 2 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 50 % phenyl methylpolysiloxane (DB 1701), 1.0 µm film thickness.

8.0 REAGENTS

NOTE: See Appendix for safety information.

8.1 Pesticide grade, or equivalent, solvents and reagents shall be used in all tests, unless otherwise indicated. Reagent water shall be organic-free. All references to water in this method refer to Millipore water from lab 211S.

8.2 Solvents - as appropriate for AC G -3510, AC G -3540, AC G -3550, AC G -3620, AC G - 3640, or AC G -3660 - n-hexane, methylene chloride, acetone and iso-octane (2,2,4-trimethylpentane). Each lot of solvent should be determined to be phthalate free. All samples and standards must be in iso-octane prior to analysis.

8.3 Stock standard solutions

8.3.1 All standards are bought as pre-made solutions that have been tested by the manufacturer. These solutions are then diluted to the required concentration with iso-octane. The solutions are purchased from Accustandard, Restek, and Ultra. Spiking solutions are purchased from different manufacturers than the solutions used for standard curves (i.e., the initial calibration standards). All solutions are purchased with quality control paperwork for the given solutions.

8.3.2 Once the vials have been opened, transfer the stock standard solutions into bottles with Teflon-lined screw caps. Store at 4 ±2°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.3.3 Stock standard solutions must be replaced before the manufacturer's expiration date, or sooner if comparison with check standards indicates a problem.

8.4 Calibration standards

8.4.1 A standard containing a mixture of Aroclor 1260 and Aroclor 1016 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 six 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

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standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors.

8.4.2 Calibration standards, at a minimum of six concentrations, are prepared through dilution of the stock standards with iso-octane and stored at room temperature. One of the concentrations should be at a concentration near, but above, the practical detection limit (PQL). The remaining concentrations should correspond to the expected range of concentrations found in real samples and should define the working range of the GC. All calibration solutions contain the injection standard.

8.4.3 Calibration solutions must be replaced before the manufacturer's expiration date of the stock, or sooner, if comparison with check standards indicates a problem.

8.4.4 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 have been used to demonstrate linearity of the detector, these single standards of the remaining five Aroclors are also used to determine the calibration factor for each Aroclor. The concentrations should correspond to the mid-point of the linear range of the detector (i.e., the same level as the mid-standard of the Aroclor 1260/1016 mixture). If Aroclors other than 1016, 1242 or 1260 are detected, a five-point calibration curve of the detected Aroclor is analyzed and the samples re-quantified against this new calibration curve.

8.5 Internal standards

The analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Isodrin is used as the internal standard. This internal standard is used to check the injection of the sample. All of the calibration standards, instrument blanks and samples have this added. The peak height must be within 80 to 120 % recovery for the injection to be deemed as a good injection. Poor injection indicates that the Teflon plug may need to be replaced or that the needle is partially plugged. A 10.0-uL spike (5000ng/µL of isodrin) is added to the GC vial containing 1.0 mL of the sample to be analyzed. Isodrin has been demonstrated not to interfere with the PCBs or the Aroclor pattern.

8.6 Surrogate standard: The analyst should monitor the performance of the extraction, clean-up (when used), analytical system, and the effectiveness of the method in dealing with each sample matrix, by spiking each sample, standard, and water blank with pesticide surrogates. Because GC/ECD analyses are more subject to interference than GC/MS analysis, a secondary surrogate is to be used when sample interference is apparent. Decachlorobiphenyl is the primary surrogate, with tetrachloro-xylene as the secondary surrogate. Proceed with corrective action when the surrogate is out of limits for a sample.

9.0 PROCEDURE

NOTE: See Appendix for safety information.

9.1 Extraction

9.1.1 Water samples are extracted with methylene chloride as a solvent using a separatory funnel (AC G -3510). Soil/sediment samples are extracted with methylene chloride ultrasonic extraction (AC G -3550) procedures. Tissue, pulp and paper samples are extracted with

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methylene chloride soxhlet extraction (AC G -3540). Oil samples are diluted with iso-octane to 10-ml final volume (AC G -3580).

9.1.2 Spiked samples (matrix spikes) and spiked blanks (lab control spikes) are used to verify the applicability of the chosen extraction technique to each new sample type. Each sample must be spiked with the compounds of interest to determine the percent recovery and the limit of detection for that sample.

9.1.2. a. Each extraction batch of samples will have the following QC:

1. Matrix spike/matrix spike duplicate of samples will be done at the rate of 10% (1 set out of 10) for regulatory samples and 5% (1 set out of 20) for all other samples.

2. All batches will include an LCS and a Method blank.

b. Spiking of water samples are performed by adding appropriate amounts of Aroclor 1016/1260 mixture (dissolved in methanol), to the water sample immediately prior to extraction. After addition of the spike, mix the samples manually. Typical spiking concentrations for water samples are 1.0 µg/L for samples in which Aroclors were not detected and 2 to 5 times the background concentration in those cases where Aroclors are present.

c. Spiking of soil, sediment and tissue samples are performed by adding appropriate amounts of Aroclor 1016/1260 mixture (dissolved in methanol), to the solid sample aliquot. The solid sample should be wet prior to the addition of the spike (at least 20% moisture) and should be mixed thoroughly with a metal spatula to homogenize the material. An appropriate amount (just enough to absorb the moisture) of sodium sulfate is added to the sample. This is thoroughly mixed. **Allow the sample to solidify for 1 hour at room temperature prior to extraction**. The sample is mixed to break up the solidified sodium sulfate to give the sample a granular/dry consistency. Additional sodium sulfate may need to be added to get this consistency. Proceed with the ultrasonic extraction (AC G -3550).

9.2 Clean up/Fractionation

9.2.1 If a sample is of biological origin, an oil, a soil/sediment sample, or contains high molecular weight materials, the use of sulfuric acid clean-up (AC G -3665) followed by florisil cartridge clean up (AC G -3620) is employed.

9.2.2 If both PCBs and pesticides are to be measured in the sample, the sample is split prior to pesticide analysis. The sulfuric acid cleanup will destroy some of the organochlorine pesticides.

9.2.3 Cleanup by AC G -3620 (unless the samples are known, or suspected to be, sampled from transformers – these samples are first analyzed at a high dilution before being subjected to florisil cleanup).

9.2.4 Elemental sulfur, which may appear in sediments and industrial wastes, interferes with the electron capture gas chromatography of the Aroclors. Sulfur should be removed by the technique described in AC G -3660, Sulfur Clean up, using activated copper.

9.3 Gas chromatography conditions (recommended)
Wide-bore columns (total runtime 12.0 minutes)
Carrier gas (He) = 10 mL/min (8.0psi constant flow at 150ºC)

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Makeup gas (argon / 5 % methane) = 100 -150 mL/min
Injector temperature = 200 °C
Detector temperature = 300 °C
Initial temperature = 150 °C, hold 0 minute
Temperature program = 150°C to 280°C at 15.0 °C/min
Final temperature = 280 °C, hold 3.33 min.

9.4 Calibration

9.4.1 Refer to AI G -8000 for proper calibration techniques. Table 2 is used for the lowest point on the calibration curve. If the laboratory is requested to use MDL, then Table 1 is used for the lowest point on the calibration curve.

9.4.2 The procedure for internal calibration is used unless there is interference from the sample. Refer to AI G -8000 for a description for this procedure.

9.4.3 The initial calibration consists of two parts:

a. A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. This six-level calibration curve of Aroclor 1016/1260 demonstrates the linearity of the detector and that the sample does not contain peaks that represent any one of the Aroclors.

b. 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 is linear).

9.5 Gas chromatographic analysis

9.5.1 Refer to AI G -8000. Add 10.0 µL of internal standard (i.e. injection standard) to the sample extract prior to injection.

9.5.2 Follow AI G -8000 for instruction on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Analysis of a mid-concentration standard after each group of 10 samples is recommended (20 samples maximum if the samples are not regulatory samples).

9.5.3 Examples of GC/ECD chromatograms generated by instruments with wide-bore columns are presented in Figures 1 through 6.

9.5.4 The sample volume injected and the resulting peak sizes (in peak heights) are recorded via the Chemstation software and transferred to the Chemserver for processing via Target software.

9.5.5 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted by the context in which the result is to be used.

9.5.6 If the peak response exceeds the working range of the system, dilute the extract and re-analyze.

9.5.7 Identification of mixtures is based on the characteristic "fingerprint" retention time and shape of the indicator peak(s). Quantitation is based on the height of the characteristic peaks as

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compared to the height of the corresponding calibration peak(s) of the same retention time and shape.

9.5.8 At least three peaks, preferably five, are used to quantitate the Aroclors. The peaks must be characteristic of the Aroclor in question. The peaks chosen are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor (Aroclor 1232, 1242, and 1016 are so similar, this is not possible). For Aroclor 1016/1260 mixture, since there is no overlap of the patterns, five peaks from each pattern are used for quantitation.

9.5.9 Late-eluting peaks are generally the most stable in the environment; therefore when it is possible, later eluting peaks in the chromatogram are selected for quantitation, pattern recognition purposes. In the case of oil samples, the later peaks may exhibit dampening of the response (probably from the background hydrocarbon interference with the ECD).

9.5.10 Identify compounds in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The retention time window used to make identifications is based upon measurements from multi-lab analysis (supplied by the EPA). Because of the stability of megabore columns, the window will never be greater than 0.07 min. In the case of oil samples, the chromatogram retention time may shift for all components. Dilutions of these samples may correct this problem.

9.5.11 If compound identification or quantitation are precluded due to interference (e.g., broad, rounded peaks or ill-defined baseline are present) clean up or dilution of the extract is warranted. It is necessary to run instrument blanks, followed by midpoint calibration checks, to determine if the instrument's integrity has been affected by the sample before further sample analysis can be continued. Refer to AC G -3600 for the procedures to be followed in sample clean up. If a sample is suspected of being high concentration or high background, multiple instrument blanks are added after the sample. These instrument blanks serve to demonstrate that there is no carryover into the next analyzed sample.

9.5.12 Each sample analysis must be bracketed by an acceptable initial or continuing calibration. When the acceptable criteria are not met, all samples must be re-analyzed under valid criteria with the following exception. If the standard analyzed after a bracketed set of samples exhibits a response for an analyte that is above the acceptance limit (i.e.; >15 %) and the analyte was not detected in the bracketed samples, then those samples do not need to be re-analyzed.

a. Verify calibration during each 12-hr shift of injection of an instrument blank followed by mid-concentration calibration of the Aroclor 1016/1260 mixture. This continuing calibration (CC) standard must also be injected after each group of 10 samples (20 samples maximum if the samples are not regulatory samples) in the analysis sequence as a calibration check. The response factors for the mid-concentration calibration should be within 15% of the average values for the multi-concentration calibration.

$$\%\,\text{Difference} = \frac{\text{average response factor - response factor of CC}}{\text{average response factor}} \times 100$$

b. When this continuing calibration is out of this acceptance window (except if the samples preceding the continuing do not contain the target analytes and the response of the analytes in the continuing give a higher response than the standards), the laboratory should stop analyses, clean the injector, replace the septum or change the syringe. An instrument blank and the continuing calibration standard are analyzed again to

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determine if the system needs to be re-calibrated. Analysis may be continued if the response factor is within the 15 % value. Otherwise, the system must be recalibrated.

c. Use the continuing calibration standard to evaluate retention time stability. If any of the standards fall outside their daily retention time windows, the system is out of control. If the problem cannot be corrected (i.e., changing the septum or changing the gas does not correct the problem), the initial calibration must be rerun and all samples run previous to the bad continuing calibration must be rerun.

d. Internal standards must be evaluated for acceptance. The measured height of the internal standard must be no more than 20% different from the height of the mid-calibration standard. When the internal standard peak height is outside the limit, the problem must be corrected before any further analysis is done. Samples that fall outside the QC criteria must be re-analyzed.

e. If compound identification or quantitation is precluded due to interference (i.e., broad, rounded peaks or ill-defined baselines are present), additional clean up of the extract and/or dilution of the extract is required.

9.6 Qualitative analysis

The qualitative analysis of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard by pattern recognition. Overlaying the sample chromatogram on the standards is the most visually rewarding in choosing which Aroclor that is most similar to that of the sample.

Tentative identification of an Aroclor must be confirmed using the second column.

9.7 Quantitation

9.7.1 Six sets of response factors will be generated for Aroclor 1016/1260 mixture, each set consisting of the response factors from each of the five peaks chosen for this mixture. The single standards for each of the other Aroclors will generate at least three response factors, one for each peak.

9.7.2 The response factors from the initial calibration are used to evaluate the linearity of the initial calibration. This involves the calculation of the mean response, the standard deviation and the relative standard deviation (RSD) for each Aroclor peak. If any of the other Aroclors are detected, that Aroclor must be run for at least 5 levels and the sample quantitated with the new quantitation curve.

9.7.3 Due to the single injection, dual-column analysis scheme, it is not practical to designate one column as the primary, the other as a confirmation column. Since the calibration standards are analyzed on both columns, the results from 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.

9.7.4 The amount of the identified Aroclor is calculated from the individual response factors for each of the 3 to 5 characteristic peaks and the calibration model (linear or non-linear) established from the multi-point calibration peaks of the 1016/1260 mixture. A concentration is determined using each of the characteristic peaks and then those 3 to 5 concentrations are averaged to determine the concentration of the Aroclor.

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9.7.5 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. If the purpose of the analysis is not regulatory compliance monitoring on the basis of Aroclor concentration, then it may be more appropriate to perform the analysis using the PCB congener approach. (Weyerhaeuser is not equipped to do PCB congener analysis at this time; therefore the congener approach has not been outlined in this method.)

9.8 GC/MS confirmation: Any compounds confirmed by two columns should also be confirmed by GC/MS if the concentration is sufficient for detection by GC/MS as determined by the laboratory-generated detection limits.

9.8.1 The GC/MS would normally require a minimum concentration of 10 ng/µL in the final extract for each PCB congener. GC/MS confirmation may not be used when the concentrations are below 1 ng/µL

9.8.2 The PCB extract and associated blank should also be analyzed by GC/MS as AC G-8270.

9.8.3 The confirmation may be from the GC/MS analysis of the base/neutral-acid extracts (sample and blank) if the surrogates and the internal standards do not interfere and it is demonstrated that the target analytes are stable during acid/base partitioning. However, if the compounds are not detected in the base/neutral-acid extract even though the concentrations are high enough, a GC/MS analysis of the PCB extract should be performed.

9.8.4 A QC reference sample of the compound must also be analyzed by GC/MS. The concentration of the QC reference standard must demonstrate the ability to confirm the Aroclors identified by GC/ECD.

10.0 QUALITY CONTROL

10.1 Quality control to validate sample extraction is covered in AC G-3500 and in the extraction method utilized. If extract clean-up is performed, follow the QC in AC G -3600 and in the specific clean-up method.

10.2 Initial Demonstration of Proficiency: Mandatory quality control to evaluate the analyst and GC system operation is found in AI G -8000.

10.2.1 The quality control reference sample concentrate (referred in AI G -8000) should contain the Aroclor 1016/1260 mix at 1 mg/L. A 0.5-mL volume of this concentrate spiked into 0.5 L of organic-free water will result in a sample concentration of 1 µg/L.

10.2.2 Instrument QC during analysis is documented above in Section 9.5.12.

10.2.3 Table 3 indicates the EPA's calibration and QC acceptance criteria for this method. Table 4 gives the EPA's method accuracy and precision as functions of concentration for the analytes of interest. The contents of the Tables are used to evaluate the laboratory's ability to perform and generate acceptable data by this method. Each analyst must demonstrate initial proficiency by generating data of acceptable accuracy and precision for target analysis in a clean matrix. New staff and significant changes in instrumentation must also repeat this initial proficiency demonstration.

10.3 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

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detection limit). At a minimum, this includes the analysis of QC samples including a method blank, a matrix spike, a duplicate, a laboratory control sample (LCS), and the addition of surrogates to each field sample and QC sample.

10.3.1 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 expected to contain target analytes, laboratories may use one matrix spike and a duplicate of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pairs, spiked with 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.

10.3.2 Due to the infrequent analysis of Aroclors, in-house method performance criteria have been set from 60 to 120 for % recovery. Should Weyerhaeuser do a frequent analysis (i.e. around 10 samples per month), the guidance found in section 6.4 of Method AI G-8000 will be used to evaluate method performance and establish performance criteria.

10.3.3 A Laboratory Control Sample (LCS) is included with each analytical batch. The LCS is a spiked clean sample (Millipore water for water extractions, pre-muffled sodium sulfate for soil/sediment extractions) with the target analytes of interest. This spike is the same as the matrix spike. When the results of the matrix spike indicate a problem due to the sample matrix, the LCS results are used to validate the extraction method for that batch.

10.3.4 All samples and QC are spiked with surrogates. Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits (limits established by performing QC procedures outlined in AI G-8000). If recovery is not within limits, the following are required:

a. Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

b. Recalculate the data and/or re-analyze the extract if any of the above checks reveal a problem.

c. Re-extract and re-analyze the sample if none of the above is a problem or flag the data as "estimated concentration".

d. Cross check the surrogate solution for correction concentration.

10.3.5 Whenever silica gel (3630) or Florisil (3620) clean-ups are used, the analyst must demonstrate that the fractionation scheme is reproducible. The LCS spike is used to characterize each batch. Recoveries must be in the range of 80% - 120%.

11.0 REPORT

11.1 Report all concentrations above the quantitation limits to 2 significant figures. Report values below the quantitation limit to 1 significant figure and flagged as “an estimate”.

11.2 PRECISION AND ACCURACY
The accuracy and precision obtainable following this method will be determined by the sample matrix, sample preparation technique, optional clean-up techniques, and calibration procedures used. The supplied accuracy and precision for water analysis from the EPA are listed in Table

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4. Table 5 shows Weyerhaeuser data for four matrix spikes for single component pesticides and four blank matrix spikes for each of the analyzed multi-component pesticides.

11.3 The MDL concentrations listed in Table 1 was obtained using organic-free reagent water. The MDL actually achievable in a given analysis will vary depending on detector response characteristics, irreducible noise from instrument electronics, and matrix effects.

12.0 KEY WORDS

AM E-8082, Aroclor, ECD, electron capture, EPA, Kuderna-Danish, KD, K-D, organochlorine, PCB, polychlorinated biphenyls

13.0 REFERENCES

13.1 U.S. Environmental Protection Agency, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846," 2nd Edition, June 11, 1991.

13.2 Organics Analysis - Multi-Media, Multi-Concentration, Document OLM 1.6, June 1991.

14.0 REVISION HISTORY

12/4/07 - Updated method procedure and method codes

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TABLE 1
Weyerhaeuser's Annual Established Method Detection Limit (MDL)
(1 mL final volume)

	µ/L	
ANALYTE	2/95 WATER DB1701	2/95 WATER DB608
Aroclor 1016	ND	ND
Aroclor 1221	ND	ND
Aroclor 1232	ND	ND
Aroclor 1242	0.016	0.012
Aroclor 1248	ND	ND
Aroclor 1254	ND	ND
Aroclor 1260	ND	ND

ND = not determined

TABLE 2
Low Concentration of the Individual Component Mixes

SEPARATE MULT- COMPONENT ANALYTES	µg/mL
Aroclor 1016/1260 Level 1	0.020
Aroclor 1016/1260 Level 2	0.040
Aroclor 1016/1260 Level 3	0.100
Aroclor 1016/1260 Level 4	0.200
Aroclor 1016/1260 Level 5	0.300
Aroclor 1016/1260 Level 6	0.400
Aroclor 1221	0.200
Aroclor 1232	0.100
Aroclor 1242	0.100
Aroclor 1254	0.100

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TABLE 3

EPA QC ACCEPTANCE CRITERIA FOR WATER ANALYSIS

Analyte	Test Conc. (µg/L)	limit for s (µg/L)	Range P,Ps (%)	Range of x (µg/L)
Aroclor 1016	50.0	10.0	50 - 114	30.5 - 51.5
Aroclor 1221	50.0	24.4	15 - 178	22.1 - 75.2
Aroclor 1232	50.0	17.9	10 - 215	14.0 - 98.5
Aroclor 1242	50.0	12.2	39 - 150	24.8 - 69.6
Aroclor 1248	50.0	15.9	38 - 158	29.0 - 70.2
Aroclor 1254	50.0	13.8	29 - 131	22.2 - 57.9
Aroclor 1260	50.0	10.4	8 - 127	18.7 - 54.9

s= Standard Deviation of four recovery measurements, in µg/L.
x = Average recovery for four recovery measurements, in µg/L
P, Ps = Percent recovery measured
D = Detected; result must be greater than zero.

Criteria from 40 CFR Part 136 for Method 608. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

TABLE 4

EPA METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATIONS

Analyte	Accuracy, as recovery, x' (µg/L)	Single Analyst precision, s' (µg/L)	Overall precision, S' (µg/L)
Aroclor 1016	0.81C+0.50	0.13x+0.15	0.15x+0.45
Aroclor 1221	0.96C+0.65	0.29x-0.76	0.35x-0.62
Aroclor 1232	0.91C+10.79	0.21x-1.93	0.31x+3.50
Aroclor 1242	0.91C+10.79	0.21x-1.93	0.31x+3.50
Aroclor 1248	0.91C+10.79	0.21x-1.93	0.31x+3.50
Aroclor 1254	0.91C+10.79	0.21x-1.93	0.31x+3.50
Aroclor 1260	0.91C+10.79	0.21x-1.93	0.31x+3.50

x' = Expected recovery for one or more measurements of a sample containing concentration C, in µg/L.
s' = Expected single analyst standard deviation of measurements at an average concentration of x, in µg/L.
S' = Expected interlaboratory standard deviation of measurements at an average concentration found of x, in µg/L.
C = True value for the concentration in µg/L.
x = Average recovery found for measurements of samples containing a concentration of C, in µg/L.

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TABLE 5

WEYERHAEUSER DATA FOR PERFORMANCE EVALUATION OF 1 Liter of Water 4/95

Analyte	Amount spike µg/L	standard deviation µg/L on DB1701	standard deviation µg/L on DB608	% recovery DB1701	% recovery DB608
PCB-1016	NA	NA	NA	NA	NA
PCB-1221	NA	NA	NA	NA	NA
PCB-1232	NA	NA	NA	NA	NA
PCB-1242	5.0	0.11	0.036	90%	83%
PCB-1248	NA	NA	NA	NA	NA
PCB-1254	NA	NA	NA	NA	NA
PCB-1260	5.0	0.024	0.017	91%	90%

Data based on two sets of four extirpations on Millipore water
NA = not analyzed

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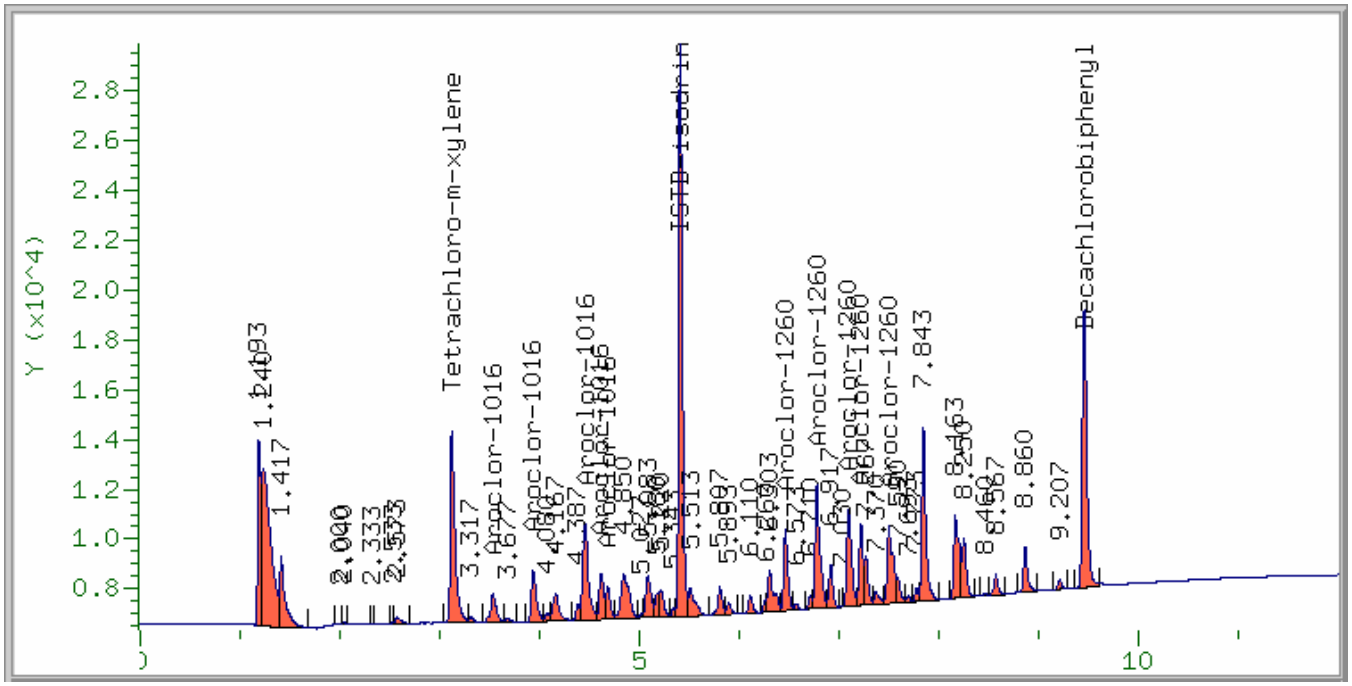

Figure 1: Aroclors 1016 and 1260 ↑

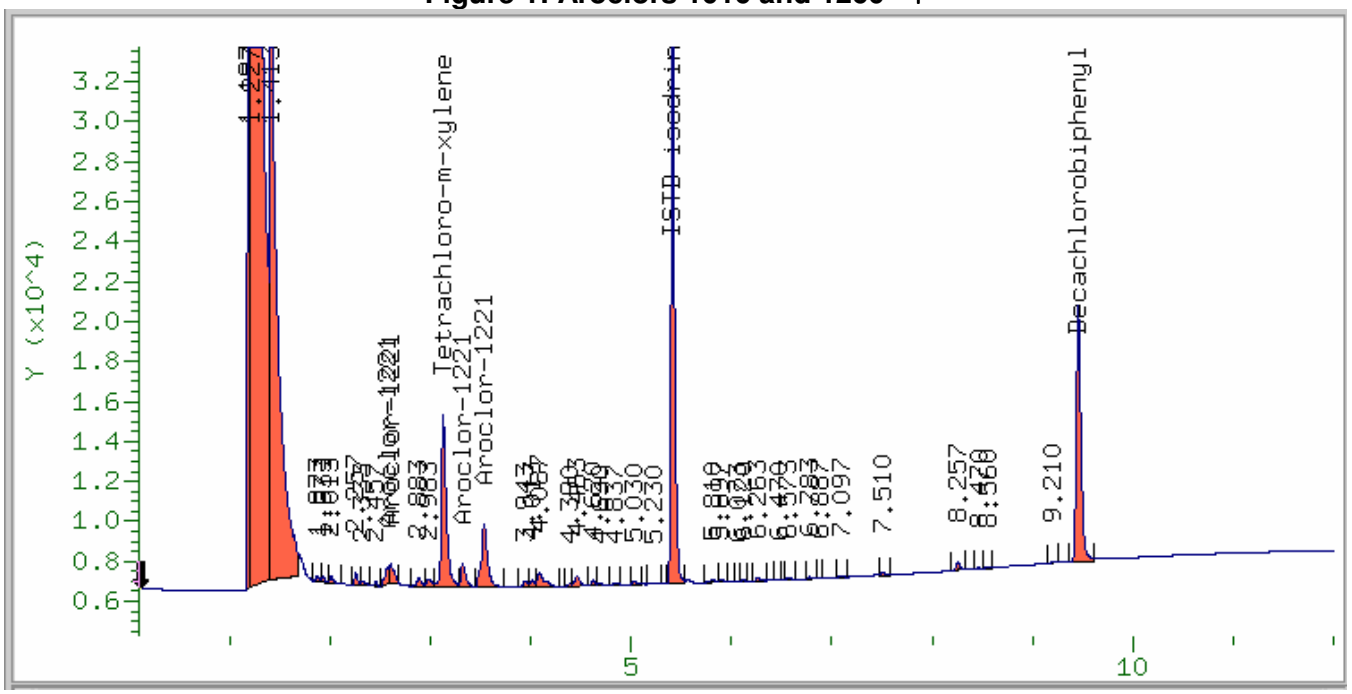

Figure 2: Aroclor 1221 ↑

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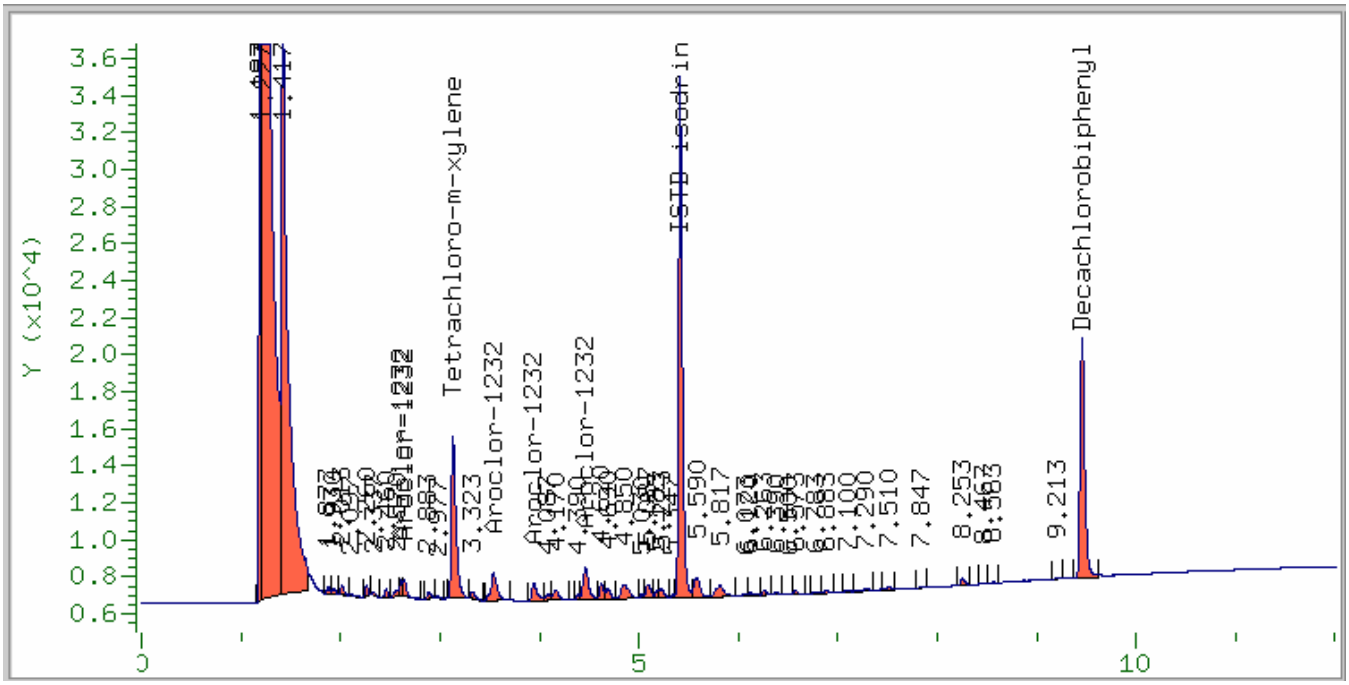

Figure 3: Aroclor 1232 ↑

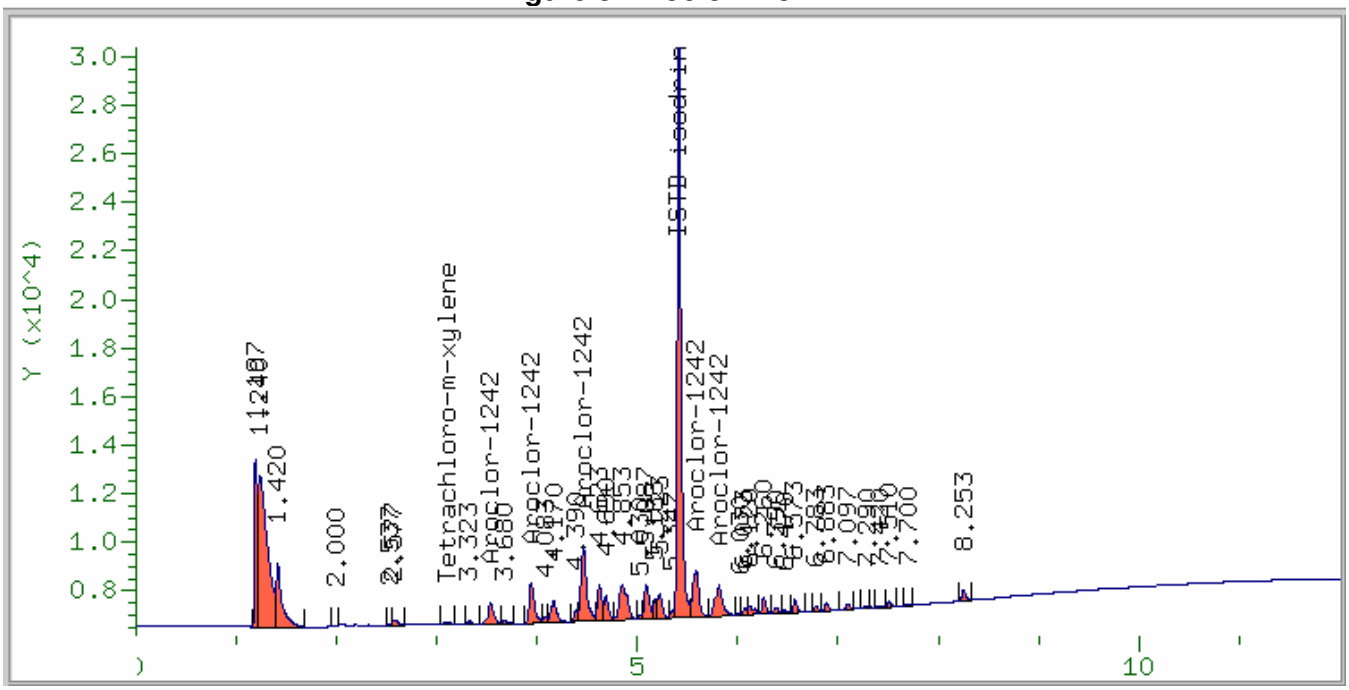

Figure 4: Aroclor 1242 ↑

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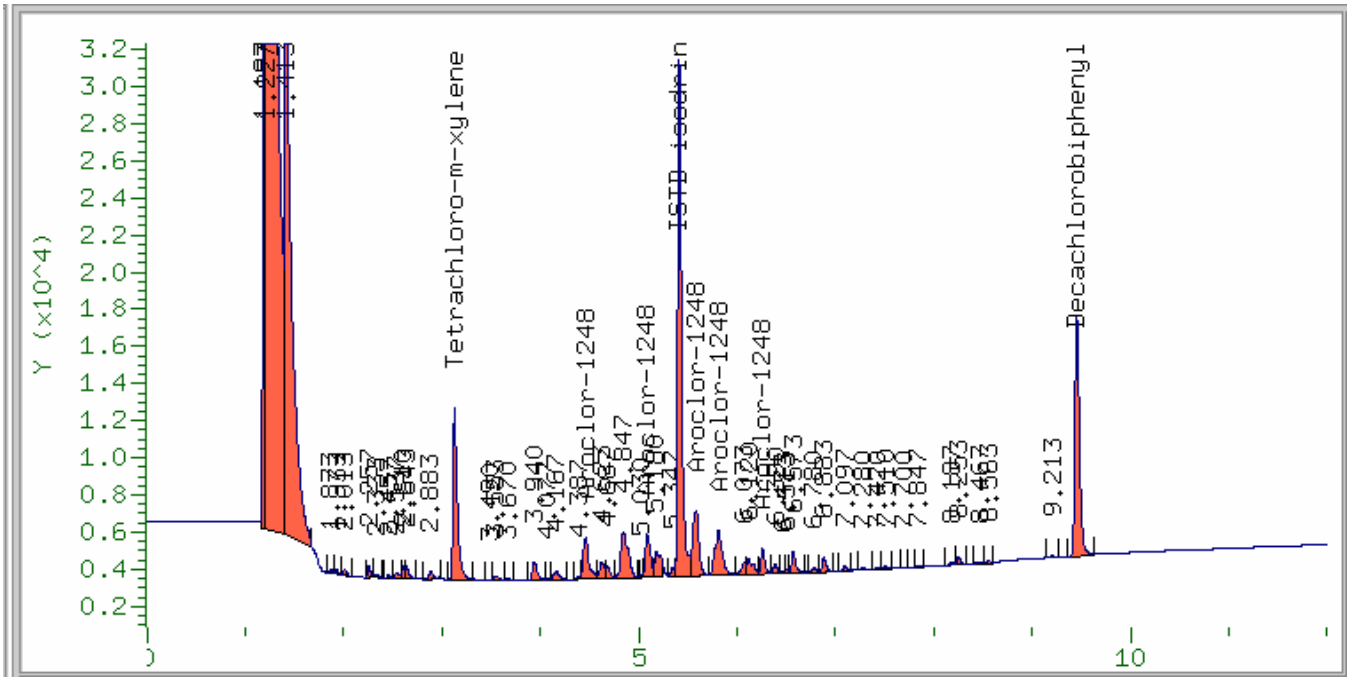

Figure 5: Aroclor 1248 ↑

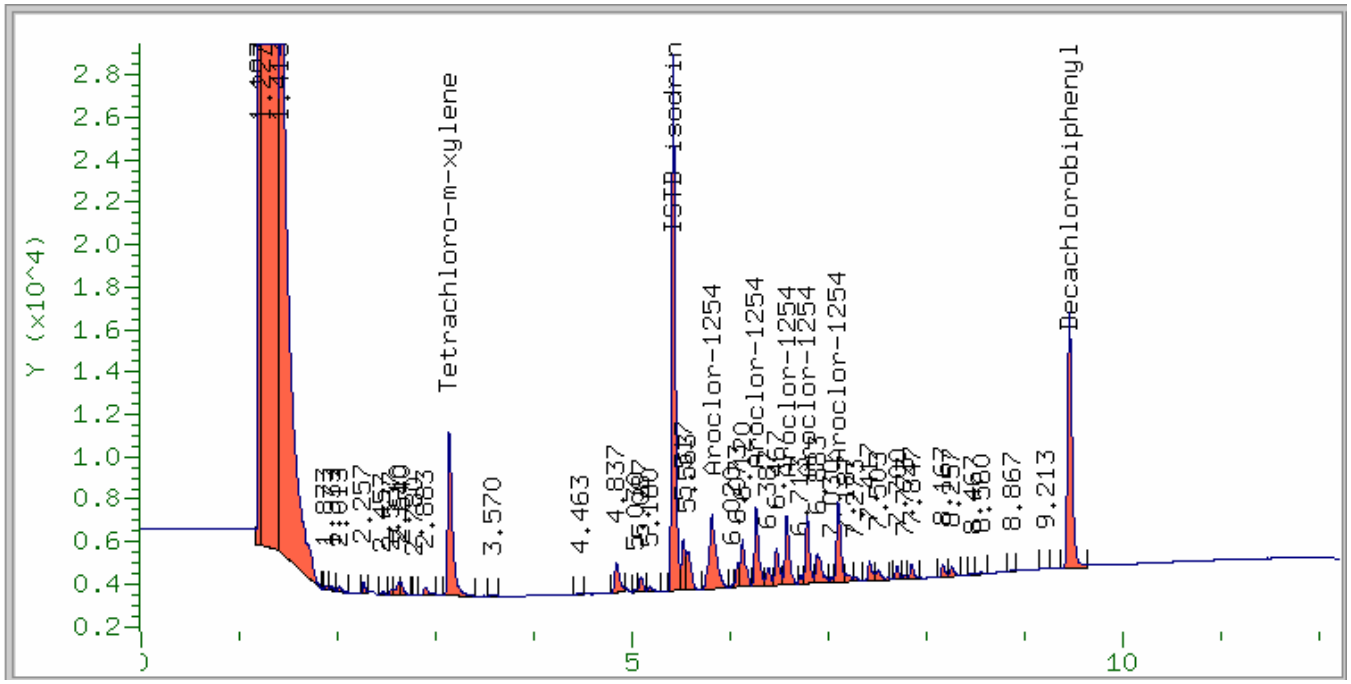

Figure 6: Aroclor 1254 ↑

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Appendix

Acetone [67-64-1]

DANGER: Acetone is highly flammable. It has a flash point of -20 °C (-4 °F) and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity).

Avoid breathing vapors. Exposure can cause coughing, dullness, headache, dizziness, nausea, irritation to the eyes and respiratory tract, narcosis, and unconsciousness. Higher concentrations may cause damage to kidneys, liver, and central nervous system (depression). Chronic exposure during pregnancy may be harmful.

Avoid skin contact. Wear neoprene gloves. Acetone is absorbed through the skin. Prolonged or repeated skin contact can cause severe irritation and dermatitis, because of the defatting action on skin. May cause redness, pain, drying and cracking of the skin. Toxicity of alcohol, and halogenated hydrocarbons may be increased.

Acetone can react vigorously with oxidizing materials. Avoid strong acids, strong alkalis, and halogens and halogen compounds.

ACGIH TWA: 500 ppm, 1,188 mg/m^3

AROCLOR STANDARDS

WARNING: Many of the compounds used to make up mixed standards are extremely toxic. The practicality of providing hazard evaluation precludes supplying hazard warnings on each substance. Also, the interactive affect of the compounds make providing an overall evaluation impossible. However, most are at very low concentrations. The standards are also handled in a manner such that the likelihood of contact is very small. The greatest hazard is probably associated with the ability of the solvent used to carry the material through the skin.

There should be little health risks if handled in accordance with safe working practices. Consult the MSDS sheet if there is one for the material.

Hexane [110-54-3]

DANGER: All manipulations involving hexane must be performed within a fume hood. Nitrile gloves are recommended. n-Hexane is highly flammable. It has a flash point of -23 °C (-9 °F), has explosive limits in air in the range of 1 - 7 %, and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity). n-Hexane can react vigorously with oxidizing materials.

Avoid breathing vapors. Exposure can cause dizziness, numbness of extremities, and intoxication. n-Hexane is a central nervous system depressant and neurotoxin. Acute exposure causes irritation, narcosis, and gastrointestinal tract irritation. Chronic inhalation causes peripheral neuropathy can have neurotoxic effects.

Avoid skin contact. n-Hexane is absorbed through the skin. Prolonged or repeated skin contact can cause irritation and dermatitis, through defatting of skin.

No PEL given.

Iso-octane (2,2,4 trimethyl pentane) [540-84-1]

WARNING: Avoid inhalation. Inhalation of iso-octane can cause mild mucous membrane irritation and central nervous system depression. Acute exposure causes irritation, narcosis, dizziness, nausea, in-coordination, stupor, unconsciousness and death.

Avoid eye contact. Liquid and high vapor concentration can cause irritation.

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Avoid skin contact. Wear nitrile gloves. Prolonged or repeated skin contact can cause irritation and dermatitis through defatting of skin. Those individuals susceptible to dermatitis should avoid handling.
Chronic exposure information is incomplete or unknown.
Avoid: strong oxidizing agents, heat, sparks, open flame, open containers, and poor ventilation.
Hazardous decomposition products: incomplete combustion can generate carbon monoxide and other toxic vapors.
Flash point: -12 °C (tag closed cup)
Flammable limits in air % by volume: lower limit: 1.0, upper limit: 6.0
TLV 350 mg/m^3 is the recommended 10-hr TWA for alkanes (C5-C8). There is presently no exposure limit established by OSHA.
Iso-octane should be protected from temperature extremes and direct sunlight. It should be stored in an acceptably protected and secure flammable liquid storage area.

Methylene chloride [75-09-2]

DANGER: All manipulations involving methylene chloride must be performed within a fume hood or a sealed system to prevent venting into the laboratory. It is non-flammable. Wear laminated film or polyvinyl alcohol (PVA) gloves.
Avoid breathing vapors. Exposure can cause anesthetic or narcotic effects, light-headedness, nausea, vomiting and headache. Excessive exposure may cause irritation to upper respiratory tract. Unconsciousness and death can result from extreme cases of over exposure. High levels may also cause cardiac arrhythmias (irregular heartbeats). Breathing vapors can elevate carboxyhemoglobin levels in the cardiovascular system thereby impairing the blood's ability to transport oxygen. Persons who smoke tobacco products will experience an intensified elevation of carboxyhemoglobin levels.
Observations in animals include liver and kidney effects.
Delayed effects: exposure may aggravate symptoms of angina (chest pains). Liver and kidney damage may occur.
Avoid skin contact. Wear PVA gloves. Prolonged or repeated exposure may cause skin irritation, even a burn. Repeated contact may cause drying or flaking of skin. Can cause irritation and dermatitis.
Cancer information: Suspect carcinogen. Causes cancer in mice and benign tumors in rats.
Avoid direct sunlight and UV sources. High temperatures and open flame may produce phosgene.
Incompatibility with metals such as: aluminum powders, potassium, sodium, and zinc powder. Avoid unintended contact with amines. Avoid contact with strong bases and strong oxidizers. Avoid prolonged contact with or storage in aluminum or its alloys.
OSHA action level: 12.5 ppm. PEL is 25 ppm. (If you can smell it, the amount is too high.)

Sodium sulfate

WARNING: Inhalation of dust may cause mild irritation to the respiratory tract.
Eye contact may cause mild irritation and redness, or mechanical irritation.
Skin contact may cause mild irritation.

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W-5

Daily Balance, Water and Temperature Checks

Process Owner (TS/PM/OM/LM) **Christine Devine**	
	Reviewed July 7, 2003 by Christine Devine Reviewed February 21, 2007 by Christine Devine
Electronic only unless colored "Controlled" stamp is present	

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1.0 SCOPE

1.1 This Standard Operating Procedure (SOP) contains the guidelines and protocols concerning daily balance, water, and temperature checks.

2.0 SUMMARY OF SOP

For each analytical laboratory, all balance, temperature, and water quality checks will be recorded daily in designated binders. The forms used for these records are controlled documents available on the Panagon document server.

3.0 PROCEDURE

3.1 All balances in the laboratory will be checked daily with a standard mass (weight) and the results recorded on quarterly Logsheets called "Daily Check Log".

3.1.1. The weights used daily are Class S. These daily masses are verified annually against a certified set of Class S Weight masses by the process owner or designee.

3.1.2. The weight used for the day is selected from the available weights and appropriate for the balance being checked.

3.1.3. The mass of the weight must be within the parameter listed for the mass of the balance being checked unless specifically stated otherwise in the appropriate method. (See Table I In attached appendix).

3.1.4. Any balance failing criteria will be flagged as not useable and appropriate laboratory personnel notified. This shall be noted on the Daily Check Log in the appropriate column for that month. It is the lab personnel's responsibility to check and verify a problem and obtain repair if necessary.

3.1.5. If the notified laboratory personnel do not respond with follow up action, a corrective action form shall be filled out and turned in to the QA advisor.

3.1.6. If several balances show a problem with a given weight, the supervisor will be contacted and the weight set will be recalibrated.

3.1.7 Once per quarter, each laboratory is required to do a Linearity Check on each balance using three different weights. These checks are noted on the bottom of each Daily Check Log. At the end of the quarter the Daily Check Log sheets are given to the process owner/designee. The process owner/designee will ensure accuracy, witness, sign and date the documents. The process owner will file away the log sheets in a three ring binder for that

year.

3.2. The temperatures of all refrigerators, incubators, freezers, and ovens will be recorded on quarterly log sheets called “Daily Check Log”.

3.2.1. A calibrated thermometer will be kept within each refrigerator, incubation, freezer and oven used for storage of samples, extractions, and standards. Replacement thermometers will be obtained at the end of each calendar year from an ISO-approved vendor.

3.2.2 The true temperature must be within the tolerance listed in Table I in the attached appendix.

3.3.3. Any temperature controlled device (in See 3.2.1) failing criteria will be flagged as not useable and appropriate laboratory personnel notified. This notification shall be noted on the Daily Check Log in the appropriate column for that month. It is the laboratory personnel’s responsibility to check and verify a problem and obtain repair if necessary.

a. Immediately notify appropriate laboratory personnel, Sample Management personnel and the Technical Specialist of any temperature-controlled unit outside of its criteria.

3.2.3. If the notified laboratory personnel does not respond with follow up action, a corrective action form shall be filled out and turned in to the QA advisor.

3.2.4 At the end of the quarter, the Daily Check Log sheets are given to the process owner/designee. The process owner/designee will witness, sign and date the documents to assure accuracy. The process owner will file away the log sheets in a three ring binder for that year.

3.3 The resistance of the Millipore Water Filtration systems will be checked and recorded on quarterly log sheets called “Daily Check Log”.

3.3.1. Water will run through the system for a minimum of 30 seconds before the resistance will be read on the built-in ohm meter.

3.3.2 The resistance shall exceed the control limit listed in Table I in the attached appendix.

3.3.3. Any system failing its criteria will be flagged as not useable and the appropriate laboratory personnel notified. This notification shall be noted on the daily check log sheet in the appropriate column for that month. It is the laboratory personnel’s responsibility to maintain their system, check and verify a problem, and obtain repair if necessary.

3.3.4 If the notified laboratory personnel do not respond with follow up action, a corrective action form shall be filled out and turned in to the QA advisor.

3.3.5 At the end of the quarter, Daily Check Log sheets are given to the process owner/designee. The process owner/designee will witness, sign and date the documents to assure accuracy. The process owner will file away the log sheets in a three ring binder for that year.

3.4 The conductance of the distilled water system will be checked and recorded on the quarterly Log sheets called “Daily Check Log”.

3.4.1 The conductance of freshly collected distilled water from the siphon tube will be read on the low level conductivity meter.

3.4.2 The conductance shall not exceed the control limit listed in Table I in the attached appendix.

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3.4.3 Any system failing its criteria will be flagged as not useable and the appropriate laboratory personnel notified. This notification shall be noted on the Daily Check Log sheet in the appropriate column for that month. It is the laboratory personnel's responsibility to repair and maintain their system.

3.4.4 If the notified laboratory personnel do not respond with follow up action, a corrective action form shall be filled out by the above and turned in to the QA advisor.

3.4.5 At the end of the quarter, the Daily Check Log sheets are given to the process owner/designee. The process owner will file away the log sheets in a three ring binder for the year.

3.5 The Table in the attached appendix is reviewed annually and updated as necessary to account for changes in calibrations, requirements, etc. All updates are to be kept within this method file.

4.0 KEYWORDS

refrigerator, incubator, freezer, oven, balance, thermometer, Millipore, filtration, distilled water

5.0 REVISION HISTORY

2/21/07 – Updated section 3.1.1, 5, 7; 3.2.1, 4; 3.3.4; and 3.4.4.

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Jun 1, 2007

Warning Limits for Class S Weight Balance Checks

Balance Range	0.1 g	0.5 g	1 g	2 g	5 g	10 g	20 g	50 g	100 g
1			1 ± 0	2 ± 0	5 ± 0	10 ± 0	20 ± 0	50 ± 0	100 ± 0
0.1		0.5 ± 0.0	1.0 ± 0.0	2.0 ± 0.0	5.0 ± 0.0	10.0 ± 0.0	20.0 ± 0.1	50.0 ± 0.1	100.0 ± 0.1
0.01	0.10 ± 0.00	0.50 ± 0.00	1.00 ± 0.00	2.00 ± 0.00	5.00 ± 0.00	10.00 ± 0.01	20.00 ± 0.01	50.00 ± 0.01	100.00 ± 0.01
0.001	0.099 ± 0.000	0.500 ± 0.000	1.000 ± 0.000	2.000 ± 0.000	5.000 ± 0.001	10.000 ± 0.001	20.000 ± 0.001	50.000 ± 0.001	99.999 ± 0.001
0.0001	0.0993 ± 0.0001	0.5000 ± 0.0001	1.0000 ± 0.0001	2.0000 ± 0.0001	5.0000 ± 0.0001	9.9998 ± 0.0001	19.9999 ± 0.0001	49.9997 ± 0.0002	99.9992 ± 0.0003
0.00001	99.31 ± 0.03	499.97 ± 0.03	1000.00 ± 0.03	1999.98 ± 0.06	4999.97 ± 0.06	9999.84 ± 0.08	19999.86 ± 0.08	49999.68 ± 0.12	99999.16 ± 0.25
0.000001	99.310 ± 0.025	499.972 ± 0.025	1000.000 ± 0.025			9999.840 ± 0.074			

If balance check is out of range -

1 Clean, rezero and recheck weight

2 If still out of range -

Flag balance, flag data, notify that laboratory's personnel

The weights for ranges 0.00001 and 0.000001 are in mg.

Note: Any 4-place or smaller balance used without a pedestal must meet limits for balance with 1 fewer place.

	0.1 g	0.5 g	1 g	2 g	5 g	10 g	20 g	50 g	100 g
Troemner	-0.010	0.018	0.01	0.01	0.00	0.00	0.04	0.02	0.14
Fisher	0.7	0.01	-0.01	0.01	0.03	0.16	0.1	0.3	0.7

Control Limits for Sample and Standard Refrigerators

4 ± 2 Degrees C

Control Limits for BOD Incubators

20 ± 1 Degrees C

Control Limit for Freezers

< -15 Degrees C

If out of range, immediately notify Laboratory and Sample Recieving Personnel

Control Limits for Millipore Water Systems

Metals Lab	> 12 megaohm
Organics Lab	> 10 megaohm

Control Limits for Distilled Water System

Conventional Lab	< 1 microSeimens/cm

If out of range, flag outlet valve and immediately notify that Laboratory's personnel

Jun 1, 2007

Laboratory	Refrigerators	Freezers	Ovens	Incubators
212 S	R10		Precision	Freas 815 Hot Pack
212 N	R		Precision	
203 S	R		Oven	
225 S			Oven #1 Oven #2 Oven #3 Oven #4 Oven #5 Oven #6 Oven #7	
227 S	R 1 R 2	F 1		
211 S	R R R R R R	 F		
233 S	R R	F F		
Cold Room	R			

W-6

General Procedures for Performing Chromatographic Separations and Calibrations

Process Owner (TS/PM/OM/LM) **Randy Eatherton**	
Assignment	Reviewed by Christine Devine July 8, 2003 Reviewed by Christine Devine September 11, 2007
Electronic only unless colored "Controlled" stamp is present	

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1.0 SCOPE AND APPLICATION

1.1 This procedure is not determinative, but instead provides guidance on analytical chromatography, describing resolution, calibration, and quality control requirements that are common to all SW-846 chromatographic procedures. AI G-8000 is to be applied in conjunction with the determinative chromatographic procedures.

1.2 Gas chromatography (GC) is a quantitative analytical technique useful for organic compounds capable of being volatilized without being decomposed or chemically re-arranged.

1.3 This procedure follows EPA method 8000B.

2.0 SUMMARY OF PROCEDURE

Each SW-846 organic analytical method provides a recommended technique for extraction, cleanup, and occasionally, derivatization of the samples to be analyzed. Before the prepared sample is introduced into the GC, a procedure for standardization must be followed to determine the recovery and the limits of detection for the analytes of interest. Following sample introduction into the GC, analysis proceeds with a comparison of sample values with standard values. Quantitative analysis is achieved through integration of peak area (for GC/MS) or measurement of peak height (for GC).

3.0 INTERFERENCES/CHROMATOGRAPHIC PERFORMANCE

3.1 Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carry-over, the sample syringe or purging device must be rinsed out between samples with water or solvent. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a solvent blank or of water to check for cross-contamination. For volatile samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide concentrations, it may be necessary to wash out the syringe, or purging device, with a detergent solution; rinse it with water; and then dry it in a 105 ± 5 °C oven between analyses.

3.2 In addition to carry-over of compounds from one sample to the next, the analysis of high concentration samples can lead to the contamination of the analytical instrument. This is particularly true to GC/MS and to GC/ECD. Eliminating this contamination can require significant time and effort in cleaning the instruments. This can be overcome by prescreening samples that are suspected or known to have high levels of organics.

3.3 One of the most important measures of chromatographic performance is resolution, the separation of chromatographic peaks. Baseline resolution of each target analyte from co-extracted materials is the goal of analytical chromatography. The ability of resolving individual compounds is generally

the limiting factor for the number of analytes that can be measured using a single procedure. Methods utilizing mass spectrometry for detection are affected less by resolution problems because overlapping peaks may still be mass-resolved.

3.4 Elevated chromatographic baselines should be minimized or eliminated during these analyses. Baseline humps can be reduced or eliminated by appropriate sample clean-up, extraction, dilution or instrument maintenance.

3.5 Poor GC performance may be expected whenever a chromatographic system is contaminated with high boiling materials. Analysis should perform routine maintenance including replacement of septa, cleaning and/or replacing the injector liners, and removing a section of the column (or retention gap if used) at the inlet end.

Capillary columns are reliable and fairly rugged, but several rules-of-thumb are required to insure column life:

3.5.1 Columns should not have contact with the oven walls (this can cause hot spots) and can weaken the column coating due to friction.

3.5.2 Oxygen kills columns. Cool the oven before changing septa, doing maintenance of the injection port or changing of columns. Flush the system with carrier gas at least 15 min before increasing the oven temperature. Inspect the injector with a leak-detector-wand and keep track of carrier gas usage. High usage indicates a leak in the system.

3.5.3 The use of guard columns (retention gaps) can increase column life by minimizing the loss of resolution when cutting off the inlet side of the column.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph - Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. All systems are equipped with data systems.

This lab has the following GC systems for which this procedure may apply:

- 3 GC/MS systems for volatiles
- 2 GC/MS systems for semivolatiles (BNA's) and Resin Acids
- 1 GC/FID/NPD system with pyrolysis unit for fingerprinting.
- 1 GC/MS system for Dioxin
- 3 GC/ECD dual column/dual detector systems for pesticides, herbicides, chlorophenolics, chlorinated hydrocarbons
- 1 GC/FID system for TPH-D and 604/608 with dual detectors/dual columns
- 1 GC/FID system for Methanol 94.03 and 99.01
- 1 GC/FID/FPD/Purge& Trap system for TPH-G, Methanol, Sulfur and Organophospate compounds
- 2 GC/FID/FPD systems for Sulfur, Organophospate compounds and Methanol
- 1 GC/TCD detector for Gas compounds
- 1 NPD detector for Organonitrogen compounds

4.2 Gas chromatographic columns - See the specific determinative method. All analyses are done with either capillary or megabore columns.

4.2.1 Narrower columns have better resolution but lower capacity and increased retention times.

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4.2.2 Longer columns can resolve more analytes, where resolution increases as a function of the square root of column length.

4.2.3 Increasing the film thickness increases column capacity, but also increases retention times.

4.2.4 The data system consists of each instrument being connected directly to a dedicated computer for data acquisition. For the GC/MS systems, the computer also controls the auto samplers. These computers are networked to a central UNIX system via LAN. Data reduction, reporting, and archiving are accomplished with TARGET and ENVISION software.

5.0 PROCEDURE

5.1 Extraction - Adhere to those procedures specified in the referring determinative method. General guidance on semivolatile extraction can be found in AC G-3500.

5.2 Clean up and separation - Adhere to those procedures specified in the referring determinative method. General guidance on cleanup procedures can be found in AC G-3600.

5.3 The recommended gas chromatographic columns and operating conditions for the instrument are specified in the referring determinative method.

5.4 Calibration - Establish gas chromatographic operating parameters equivalent to those indicated in the determinative method of interest. Prepare calibration standards using the procedures indicated in the determinative method of interest. Calibrate the chromatographic system using either the external standard technique or the internal standard technique.

5.4.1 External standard calibration procedure

a. For each analyte of interest, prepare calibration standards at a minimum of five concentrations by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with an appropriate solvent. The lowest of the external standards should be the method quantitation limit (based on a concentration with no dilution). The other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. At least one of the calibration concentrations should correspond to a sample concentration at or below any regulatory or action limits associated with a target compound.

b. Inject each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph (e.g., 1 - 5 µL on column, 5 mL with purge-and-trap). Tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each analyte. Alternatively, for samples that are introduced into the gas chromatograph using a syringe, the ratio of the response to the amount injected, defined as the Calibration Factor (CF), can be calculated for each analyte at each standard concentration. If the percent relative standard deviation (% RSD) of the calibration factor is less than 20 % (this may vary according to different methods) over the working range, linearity through the origin can be assumed. In such cases, the average calibration factor can be used in place of a calibration curve.

$$\text{Calibration factor} = \frac{A_x}{C_f V_i}$$

where:

Ax = response for the analyte, units may be in area counts (for GC/MS) or peak height (for GC)

Cf = Concentration of analyte, unit is ng/µL

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Vi = Volume of extract injected, µL. For purge-and-trap analysis, Vi is not applicable and therefore = 1.

NOTE: For multi-response pesticides/PCBs, at least three of the major peaks are calculated against a separate response factor for each of those peaks. The average of these calculated amounts are then used for quantitation, i.e., concentration = (average)(concentration of major peaks).

c. The working calibration curve or calibration factor must be verified on each working day of analysis by the injection of one or more calibration standards. The frequency of verification is dependent on the detector. Detectors, such as the electron capture detector, that operate in the sub-nanogram range are more susceptible to changes in detector response caused by GC column and sample effects. Therefore, more frequent verification of calibration is necessary - every 12 hr of analysis or every 10 samples, depending on the method. The flame ionization detector is much less sensitive and requires less frequent verification. If the response for any analyte varies from the predicted response by more than ± 15 %, a new calibration curve must be prepared for that analyte.

$$\text{Percent Difference} = \frac{(R_1 - R_2)\,x\,100}{R_1}$$

where:
R1 = calibration factor from initial calibration
R2 = calibration factor from succeeding analysis

5.4.2 Internal standard calibration procedure

a. To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard applicable to all samples can be suggested.

b. Prepare calibration standards at a minimum of five concentrations for each analyte of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards and dilute to volume with an appropriate solvent. The lowest of the standards should be the method quantitation limit (based on a concentration with no dilution). The other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. At least one of the calibration concentrations should correspond to a sample concentration at or below any regulatory or action limits associated with a target compound.

c. Inject each calibration standard using the same introduction technique that will be applied to the actual samples, e.g., 1-5 µL on-column injection, 5 mL with purge-and-trap, etc. Tabulate the peak height or area responses against the concentration of each compound and internal standard. Calculate response factors (RF) for each compound as follows:

$$RF = (A_s)(C_{is})/(A_{is})(C_s)(V_{inj})$$

where:
As = response for the analyte to be measured
Ais = response for the internal standard
Cis = concentration of the internal standard, ng/µL
Cs = concentration of the analyte to be measured, ng/µL

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Vinj = Volume of extract injected in µL. For purge-and-trap analysis, Vinj is not applicable and therefore = 1.

If the RF value over the working range is constant (< 20 % RSD,) the RF can be assumed to be invariant, and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, As/Ais vs. RF

d. The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. The frequency of verification is dependent on the detector. Detectors, such as the electron capture detector, that operate in the sub-nanogram range are more susceptible to changes in detector response caused by GC column and sample effects. Therefore, more frequent verification of calibration is necessary (every 12 hr or every 10 samples, depending on the method). The flame ionization detector is much less sensitive and requires less frequent verification. If the response for any analyte varies from the predicted response by more than ± 15 % (this may vary according to different methods), a new calibration curve must be prepared for that compound.

5.4.3 Retention time windows

Retention times produced from using megabore or capillary columns are very stable. During 72 hr of analysis, it is not unusual for retention time to drift less than 0.01 min. Because of this stability in retention time, a maximum retention time window of 0.10 min has been adapted for general GC analysis (Pesticides have set windows based on lab-pooled results) and 0.50 min for GC/MS analysis. (Semivolatiles and Volatiles use RRT.) For multi-response analytes (i.e., PCBS), the analyst should use the retention time window, but should primarily rely on pattern recognition, where the experience of the analyst weighs heavily in the interpretation of chromatograms. The retention time of the initial calibration standards is used for determining retention times during the sequence.

5.4.4 Gas chromatographic analysis

a. Introduction of organic compounds into the gas chromatograph varies depending on the volatility of the compound. Volatile organics are primarily introduced by purge-and-trap (EPA Method 5030). However, there are limited applications (in EPA Method 5030) where direct injection is acceptable. Use of EPA Method 3810 or 3820 as a screening technique for volatile organic analysis may be valuable with some sample matrices to prevent overloading and contamination of the GC systems. Semivolatile organics are introduced by direct injection.

b. The appropriate detector(s) is given in the specific method.

c. Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with mid-point continuing calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded. Sample sets are always bracketed with continuing calibration checks.

All instrument calibration and check standards must be preceded by an instrument blank, to demonstrate that there is no carry-over to affect calculations. Any highly contaminated samples must be followed with instrument blanks, to demonstrate that there is no cross contamination with following samples.

d. Direct Injection. Inject 2-5 ± 0.05 µL of the sample extract using the solvent flush technique, if the extract is manually injected. Smaller volumes (1.0 ± 0.05 µL) can be injected, and the solvent

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flush technique is not required, 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.

e. If the response exceeds the linear range of the system, dilute the extract and re-analyze. It is recommended that extract be diluted so that all peaks are on scale. Optimally, the diluted concentration would place the analyte concentration near the mid-point of the calibration range. Overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure all peaks are on scale over a 100-fold range, are acceptable if linearity is demonstrated. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

f. If peak detection is prevented by the presence of interferences, further cleanup is required.

g. Examples of chromatograms for the compounds of interest are frequently available in the referring analytical method and commercial suppliers of capillary columns.

h. Calibrate the system immediately prior to conducting any analyses (see part 5.4). A mid-concentration standard must also be injected at intervals specified in the method and at the end of the analysis sequence. The calibration factor for each analyte to be quantitated, must not exceed a 15 % difference when compared to the initial calibration standards of the analysis sequence. When this criterion is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is necessary before re-calibrating and proceeding with sample analysis. All samples that were injected after the standard exceeding the criteria must be re-injected, if the initial analysis indicated the presence of the specific target analytes that exceeded the criteria.

i. Use the absolute retention time for each analyte from part 5.4.3 as the midpoint of the window for that day. The daily retention time window equals the midpoint ± 0.10 min for general GC analysis and ± 0.50 min for GC/MS analysis - unless specified differently in the given method.

j. Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention-time window. Normally, confirmation is required on a second GC column, by GC/MS if concentration permits, or by other recognized confirmation techniques. Confirmation may not be necessary if the composition of the sample matrix is well established by prior analyses.

5.4.5 Suggested chromatography system maintenance. Corrective measures may require any one or more of the following remedial actions.

a. Capillary/Megabore columns - Clean and deactivate the glass injection-port insert or replace with a cleaned and deactivated insert. Break off the first few centimeters (up to 10 cm) of the injection port side of the column. If the column is using a retention gap/guard column, remove the first few centimeters, or replace the retention gap/guard column. Remove the column 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 column.

b. Metal injector body - Usually, the split line needs flushing and deactivation. Turn off the oven and remove the analytical column when the oven has cooled. Remove the glass injection port insert (instruments with off-column injection or Grob). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

Cool the Injection Port (< 40 °C). Place a beaker beneath the injector port inside the GC oven. Using a wash bottle, serially rinse the entire inside of the injector port with acetone, methylene chloride, and then hexane; catching the rinseate in the beaker. If the problem is with the split line,

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disconnect the split line at the split solenoid, and connect a glass syringe to this end of the line. Flush with water, acetone, methylene chloride, and hexane. Sylon is not necessary.

ACETONE [67-64-1]

DANGER: Acetone is highly flammable. It has a flash point of -20 °C (-4 °F) and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity).
Avoid breathing vapors. Exposure can cause coughing, dullness, headache, dizziness, nausea, irritation to the eyes and respiratory tract, narcosis, and unconsciousness. Higher concentrations may cause damage to kidneys, liver, and central nervous system (depression). Chronic exposure during pregnancy may be harmful.
Avoid skin contact. Acetone is absorbed through the skin. Prolonged or repeated skin contact can cause severe irritation and dermatitis, because of the defatting action on skin. May cause redness, pain, drying and cracking of the skin. Toxicity of alcohol, and halogentated hydrocarbons may be increased.
Acetone can react vigorously with oxidizing materials. Avoid strong acids, strong alkalis, and halogens and halogen compound.
ACGIH TWA: 500 ppm, 1,188 mg/m^3

Hexane [110-54-3]

DANGER: All manipulations involving hexane must be performed within a fume hood. Nitrile gloves are recommended. n-Hexane is highly flammable. It has a flash point of -23 °C (-9 °F), has explosive limits in air in the range of 1 - 7 %, and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity). n-Hexane can react vigorously with oxidizing materials.
Avoid breathing vapors. Exposure can cause dizziness, numbness of extremities, and intoxication. n-Hexane is a central nervous system depressant and neurotoxin. Acute exposure causes irritation, narcosis, and gastrointestinal tract irritation. Chronic inhalation causes peripheral neuropathy can have neurotoxic effects.
Avoid skin contact. n-Hexane is absorbed through the skin. Prolonged or repeated skin contact can cause irritation and dermatitis, through defatting of skin.
No PEL given.

Methylene chloride [75-09-2]

DANGER: All manipulations involving methylene chloride must be performed within a fume hood or a sealed system to prevent venting into the laboratory. It is non-flammable. Wear laminated film or polyvinyl alcohol (PVA) gloves.
Avoid breathing vapors. Exposure can cause anesthetic or narcotic effects, light-headedness, nausea, vomiting and headache. Excessive exposure may cause irritation to upper respiratory tract. Unconsciousness and death can result from extreme cases of over exposure. High levels may also cause cardiac arrhythmias (irregular heartbeats). Breathing vapors can elevate carboxyhemoglobin levels in the cardiovascular system thereby impairing the blood's ability to transport oxygen. Persons who smoke tobacco products will experience an intensified elevation of carboxyhemoglobin levels. Observations in animals include liver and kidney effects.
Delayed effects: exposure may aggravate symptoms of angina (chest pains). Liver and kidney damage may occur.
Avoid skin contact. Prolonged or repeated exposure may cause skin irritation, even a burn. Repeated contact may cause drying or flaking of skin. Can cause irritation and dermatitis.
Cancer information: Suspect carcinogen. Causes cancer in mice and benign tumors in rats.
Avoid direct sunlight and UV sources. High temperatures and open flame may produce phosgene.

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Incompatibility with metals such as: aluminum powders, potassium, sodium, and zinc powder.
Avoid unintended contact with amines. Avoid contact with strong bases and strong oxidizers.
Avoid prolonged contact with or storage in aluminum or its alloys.
OSHA action level: 12.5 ppm. PEL is 25 ppm. (If you can smell it, the amount is too high.)

c. Prepare a solution of deactivating agent (Sil-Prep) following manufacturer's directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, serially rinse the injector body with toluene, methanol, acetone, and hexane. Re-assemble the injector, bring the injector to temperature, and allow a good amount of helium flushing of the system before replacing the GC column.

DANGER: See **DANGER** under 'acetone' in part b. above.

Methanol, [67-56-1]

WARNING: All manipulations involving methanol should be performed within a fume hood. Wear nitrile gloves and a lab coat. Methanol is flammable and burns with a clear, almost invisible flame. It has a flash point of 12 °C, explosive limits in air in the range of 6 - 36 %, and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity). Methanol can react vigorously with strong oxidizing materials. Avoid contact with strong acids, acid chlorides, acid anhydrides, and alkali metals.
Avoid breathing vapors. Initial symptoms may be only that of mild intoxication, but may become severe after 12 to 18 hours. Methanol can be irritating to mucous membranes and upper respiratory tract. Exposure can cause headache, muscle weakness, dizziness, nausea, vomiting, narcosis, respiratory failure, low blood pressure, gastrointestinal disturbances, convulsions, and may be fatal. Methanol is a central nervous system depressant and can cause kidney, liver (enlargement), eye (especially optic nerve) and heart damage.
Avoid skin contact. Methanol is absorbed through the skin. Prolonged or repeated skin contact can cause irritation and dermatitis.
Eye contact may cause irritation and/or temporary corneal damage.
Prolonged exposure to methanol has been known to cause fetal development abnormalities in pregnant rats.

US ACGIH TWA: 200 ppm; STEL: 250 ppm.

Toluene [108-88-3]

WARNING: All manipulations involving toluene must be performed within a fume hood. Wear PVA gloves. Toluene is highly flammable. It has a flash point of 4 °C, has explosive limits in air in the range of 1 - 7 %, and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity).
Avoid breathing vapors. High concentrations are extremely destructive to tissues of the mucous membranes and upper respiratory tract (irritation), eyes and skin.
Symptoms of exposure may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, dizziness, dullness, narcosis, hallucinations, distorted perceptions, changes in motor activity, headache, nausea and vomiting, diarrhea, convulsions and unconsciousness. Exposure can cause lung irritation, chest pain, and edema, which may be fatal.
Chronic effects may cause nervous system disturbances. Inhalation studies on toluene have demonstrated the development of inflammatory and ulcerous lesions of the penis, prepuce and scrotum in animals.
Avoid skin contact. Toluene is absorbed through the skin. Prolonged or repeated skin contact can cause irritation and dermatitis, through defatting of skin.
Consumption of alcohol may increase toxic effects.

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Eye contact can cause severe eye irritation and may cause temporary corneal damage.
Target organs: brain and coverings, (depression of) central nervous system, autonomic nervous system (parasympathomimetic), liver (damage), kidneys (damage), bladder and lungs.
Incompatible with strong oxidizing agents, nitric acid, sulfuric acid, chlorine.
No known PEL.

5.4.6 Calculations

a. External standard calibration - The concentration of each analyte in the sample may be determined by calculating the amount of standard purged or injected, from the peak response, using the calibration curve or the calibration factor determined in part 5.4.1. All injections in the lab are done via auto samplers and therefore all standards and samples are injected at a constant volume. Under no circumstances will samples be analyzed at different injection parameters from the standards associated with those samples. The concentration of a specific analyte is calculated as follows:

Aqueous samples

$$\text{Concentration}\,(\mu g/L) = \frac{(A_x)(V_{fv})(D)}{(C_f)(V_{inj})(V_s)}$$

where:

Ax = response for the analyte in the sample, units may be in area counts (for GC/MS) or peak height (for GC).
Vfv = final volume of total extract, µL. For purge-and-trap analysis, Vfv is not applicable and therefore = 1
D = dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D = 1, dimensionless
Cf = Calibration factor of external standard, units in response/ng
Vinj = Volume of extract injected in µL. For purge-and-trap analysis, Vinj is not applicable and therefore = 1.
Vs = volume and sample extracted or purged, mL

Non aqueous samples

$$\text{Concentration}\,(\mu g/kg) = \frac{(A_x)(V_{fv})(D)(U)}{(C_f)(V_{inj})(W)}$$

where:

Ax = response for the analyte in the sample, units may be in area counts (for GC/MS) or peak height (for GC).
W = dry mass of sample extracted or purged, g. Wet mass will only be used upon request.
U = Correction factor for pesticide analysis, where GPC cleanup looses ½ of the sample, U is 2.
Cf = Calibration factor of external standard, units in response/ng
Vinj = Volume of extract injected in µL. For purge-and-trap analysis, Vinj is not applicable and therefore = 1.
Vs = volume and sample extracted or purged, mL
Ax, Vfv, D, Cf and Vinj have the same definition as for aqueous samples.

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b. Internal standard calibration. For each analyte of interest, the concentration of that analyte in the sample is calculated as follows:

Aqueous samples

$$\text{Concentration}\,(\mu g/L) = \frac{[(A_x)][(C_{is})/A_{is}](V_{fv})(D)]}{(RF)(V_{inj})(V_s)}$$

where:

Ax = response for the analyte in the sample, units may be in area counts (for GC/MS) or peak height (for GC).
Ais = response of the internal standard, units same as Ax
Cis = amount of internal standard added to extract or volume purged, ng
D = dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D = 1, dimensionless
Vfv = final volume of total extract, µL. For purge-and-trap analysis, Vfv is not applicable and therefore = 1
Cf = Calibration factor of external standard, units in response/ng
Vinj = Volume of extract injected in µL. For purge-and-trap analysis, Vinj is not applicable and therefore = 1.
Vs = volume and sample extracted or purged, mL
RF = response factor for analyte, units are dimensionless.

Non aqueous samples

$$\text{Concentration}\,(\mu g/kg) = \frac{[(A_x)((C_{is})/A_{is})](V_{fv})(D)(U)}{(RF)(V_{inj})(W_s)}$$

where:

Ws = dry mass of sample extracted, g.
U = Correction factor for pesticide analysis, where GPC clean-up loses ½ of the sample, U is 2.

As, Cis, Vfv, D, Ais, RF, and Vini have the same definition as for aqueous samples.

6.0 QUALITY CONTROL

6.1 Refer to the specific determinative method for quality control procedures.

6.2 The experience of the analyst in performing gas chromatography is invaluable to the success of the methods.

6.2.1 Each day that analysis is performed, an instrument blank and the daily calibration sample should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are:

- Do the peaks look normal?
- Is the response obtained comparable to the response from previous calibrations?
- Are the instrument blanks clean?

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Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., column changed,) re-calibration of the system must take place.

6.2.2 The performance of the entire analytical system should be checked daily, using data gathered from analyses of blanks, standards, and replicate samples. Significant peak tailing must be corrected. Tailing problems are generally traceable to active sites on the GC column or to the detector operation.

6.2.3 The precision between replicate analyses of standards and check samples should be evaluated. A properly operating system should perform with an average relative standard deviation of less than 10 % (this may vary according to different methods). Poor precision is generally traceable to pneumatic leaks, especially at the injection port, i.e., leaking septum or loose syringe plunger.

6.3 Summary of Required Instrument QC

6.3.1 The % RSDs vary by ≤ 20 % (this may vary according to different methods) when comparing calibration factors to determine if a five-point calibration curve is linear.

6.3.2 The % RSD limit is ± 15 % (this may vary according to different methods) difference when comparing daily response of a given analyte versus the initial response. If the limit is exceeded, a new standard curve must be prepared.

6.3.3 Part 5.4.3 indicates the establishment of retention time windows.

6.3.4 The % RSDs are ± 15 % (this may vary according to different methods) difference when comparing the initial response of a given analyte versus any succeeding standards analyzed during an analysis sequence.

6.3.5 All succeeding standards in an analysis sequence must fall within the daily retention-time window established by the first standard of the sequence.

6.4 Initial Demonstration of Proficiency. To establish the ability to generate data of acceptable bias and precision, the analyst must perform the following operations:

6.4.1 A Quality Control (QC) check sample concentrate is required containing each analyte of interest. The QC check sample concentrate may be prepared from pure standard materials, or purchased as certified solutions. If prepared by the laboratory, the QC check sample concentrate must be made using stock standards prepared independently from those used for calibration.

The concentration of the QC check sample concentrate is highly dependent upon the analytes being investigated. Therefore, refer to AC G-3500, for the required concentration of the QC check sample concentrate.

6.4.2 Preparation of QC check sample: Refer to AP G-ORG STD.

6.4.3 Analyze replicate aliquots (at least four) of the well-mixed QC check sample by the same procedures used to analyze actual samples in each of the methods. For volatile organics, the preparation/analysis process is purge-and-trap/gas chromatography or direct injection/gas chromatography. For semivolatile and pesticide organics, the QC check samples must undergo solvent extraction (see AC G-3500) prior to chromatographic analysis.

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6.4.4 Calculate the average recovery in µg/L, and the standard deviation of the recovery(s) in µg/L, for each analyte of interest using the four (or more) results.

6.4.5 For each analyte compare s and x with the corresponding acceptance criteria for precision and accuracy, respectively, given in the QC Acceptance Criteria Table at the end of each of the determinative methods. In the absence of method-specific acceptance criteria for the initial demonstration of proficiency, use the recoveries of 70 - 130 % as guidance in evaluating the results. If all analytes of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual analyte exceeds the precision limit or any individual falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

NOTE: The large number of analytes in each of the QC Acceptance Criteria Tables present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are determined.

6.4.6 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to part 6.4.6.a or 6.4.6.b.

a. Locate and correct the source of the problem and repeat the test for all analytes of interest beginning with part 6.2.1.

b. Beginning with part 6.2.1, repeat the test only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest.

6.5 Matrix Spike and Laboratory Control Samples

The laboratory procedures for documenting the effect of the matrix on method performance includes the analysis of at least one matrix spike, and either one matrix duplicate or one matrix spike duplicate per analytical batch, or at the rate of 5 %, whichever is greater.

In addition, a Laboratory Control Sample (LCS) is included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same mass 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 indicates 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. The rate of analysis of the LCS is one per analytical batch.

6.5.1 The concentration of the spike in the sample should be determined as follows:

a. If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit, the spike should be at that limit.

b. If the concentration of a specific analyte in a water sample is not being checked against a limit specific to that analyte, the spike should be 2-10 times higher than the quantitation limit.

6.5.2. Analyze one unspiked and one spiked sample aliquot to determine percent recovery of each of the spiked compounds.

a. Volatile organics. Analyze one 5 ± 0.05-mL sample aliquot to determine the background concentration of each analyte. If necessary, prepare a new QC reference sample concentrate (part

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6.4.2) appropriate for the background concentration in the sample. Spike a second 5-mL sample aliquot with 10 ± 0.1 µL of the QC reference sample concentrate and analyze it to determine the concentration after spiking of each analyte. Calculate each percent recovery (%R) as:

$$\%R = \frac{100(x_s - x_u)}{K}$$

where:
xs = measured value for spiked sample,
xu = measured value for unspiked sample,
K = known value of the spike in the sample.

b. Semivolatile organics. Analyze one sample aliquot (extract of 500-mL sample) to determine the background concentration of each analyte. Spike a second 500-mL sample aliquot with 0.25 ± 0.01 mL of the QC reference sample concentrate and analyze it to determine the concentration after spiking of each analyte. Calculate each percent recovery according to the calculation in part 6.5.2a. NOTE: There may be times where insufficient sample volume exists to use 500-mL for the spike. In such cases, use the remaining sample, record the volume in the extraction logbook, and use the value to calculate xs.

6.5.3 Compare the percent recovery (% R) for each analyte in a water sample with the corresponding criteria presented in the QC Acceptance Criteria Table found at the end of each of the determinative methods. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1. If spiking was performed at a concentration lower than the QC reference sample concentration (part 6.4.2), the analyst must use either the QC acceptance criteria presented in the tables, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of an analyte: (1) calculate accuracy (x') using the equation found in the Method Accuracy and Precision as a Function of Concentration Table (appears at the end of each determinative method), substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in the same Table, substituting x' for x; (3) calculate the range for recovery at the spike concentration as [(100x'/T) ± 2.44] x (100S'/T) %. Many methods do not contain acceptance criteria for LCS results. 70 - 130 % should used as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed. When in-house limits have been developed for matrix spike recoveries, the LCS results should fall within those limits.

6.5.4 If any individual % R falls outside the designated range for recovery, that analyte has failed the acceptance criteria. A check standard containing each analyte that failed the criteria must be analyzed.

6.5.5 Compare the recovery data from the matrix spike with the LCS data (use the average recovery if a matrix spike and matrix spike duplicate were analyzed). If any individual percent recovery in the matrix spike (or matrix spike duplicate) falls outside the designated range for recovery, the analyst determines if there is a matrix effect or a laboratory performance problem. A matrix effect is indicated if the LCS data are within limits but the matrix spike data exceed the limits. The surrogate recovery data should also be used to evaluate the data. If any analyte in a water sample fails the acceptance criteria for recovery in part 6.5.3 and is determined not to be a matrix effect, a QC reference standard containing each analyte that failed must be prepared and re-analyzed.

6.6 Method Blanks

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6.6.1 Method blanks are prepared at one method blank for each batch.

6.6.2 When samples that are extracted in the same batch are analyzed on separate instruments or on separate analytical shifts, the method blank associated with those samples must be analyzed on all the instruments used for sample analysis, and on all the analytical shifts.

6.6.3 The results of the method blank should be less than ½ of the quantitation limit. There is no blank subtraction from the reported results. The results for any analyte that is also found in the method blank at levels higher than ½ of the quantitation limit will be re-extracted.* If the holding time is exceeded with the re-extraction, then both the initial extraction and re-extraction will be reported in the data package.

*** See QA Plan for exceptions to this.**

6.7 Surrogate recoveries

6.7.1 It is necessary that surrogate recovery data be evaluated from individual samples versus surrogate recovery limits developed in the laboratory. The general considerations for developing in-house acceptance criteria for surrogate recoveries are described in part 6.8.3-6.8.4.

6.7.2 Surrogate recovery is calculated as:

$$\text{Recovery (\%)} = \frac{\text{Concentration found}}{\text{Concentration added}} \text{ x } 100$$

If the recovery is not within in-house surrogate recovery limits, the following procedures are necessary:

a. Examine the chromatograms for interfering peaks. If there appears to be interfering peaks, note this in the case narrative.

b. Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the extract.

c. If no problem is found, re-extract and re-analyze the sample.

d. If upon re-analysis, the recovery is again outside of recovery limits, flag the data as "estimated concentration". If the recovery is within the limits, report the re-analysis data. If the holding time for the method has expired prior to the re-analysis, provide both the original and the re-analysis results and note the holding time.

6.8 Generating performance criteria for matrix spike recoveries, surrogate recoveries, initial demonstration of proficiency, and laboratory control sample recoveries. (See QA Plan for exceptions to this. Note also 6.8.6.)

The laboratory is in the process of setting up of automated calculation of criteria for matrix spike recoveries, surrogate recoveries, and laboratory control sample (LCS) recoveries. Along with this will be the automated generation of control charts for laboratory control sample recoveries and method blank surrogate recoveries. At present, the recovery criteria for all GC and GC/MS methods are manually being generated, except in the case of AM E-608, -8081 and -8082, where infrequent analysis does not generate enough data for control charts. These methods that are done infrequently have limits derived from the SW-846 methods.

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The procedure for the calculation of performance criteria is:

6.8.1 Calculate the average percent recovery and standard deviation for each of the matrix spike compounds after analysis of 20 matrix spike samples of the same matrix.*

*** See QA Plan for exceptions to this. Note also 6.8.6.**

6.8.2 Calculate the upper and lower control limit for each matrix spike or surrogate compound:

Upper Control Limit = x + 3s

Lower Control Limit = x - 3s

6.8.3 Calculate the upper and lower warning limit for each matrix spike or surrogate compound:

Upper Warning Limit = x - 2s

Lower Warning Limit = x - 2s

6.8.4 Any matrix spike or surrogate results outside of the control limits require corrective action by the laboratory, including but not limited to, the review of the sample results, inspection of the chromatographic system and re-analysis of the samples.

The warning limits should be used to guide internal evaluations of method performance, track the performance of individual analysts, and monitor the effects of change to the analytical procedures. Repeated results outside of the warning limits should result in corrective actions.

6.8.5 Once established, control limits and warning limits for matrix spike compounds should be updated after every 20 matrix spike samples of the same matrix* or at least quarterly. Control limits and warning limits for surrogates should be updated after every 20 field samples of the same matrix or at least quarterly. Trends should be tracked in both performance and in the control limits themselves.

*** See QA Plan for exceptions to this. Note also 6.8.6.**

6.8.6 For methods and matrices with very limited data (i.e., unusual matrices not analyzed often), interim limits are established using available data (i.e., the method QC criteria) or by analogy to similar methods or matrices.

6.8.7 Results used to develop acceptance criteria should meet all other QC criteria associated with the determinative method.

6.8.8 Because of the large variability of matrix types, the method blanks and LCS surrogate recoveries will be used to establish surrogate recoveries for those matrices with which they were extracted.

6.9 Additional quality assurance programs are practiced in the lab. As mentioned above, LCS are analyzed with every batch at the rate of at least 5 %. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer are used. The laboratory analyzes standard reference materials (through semi-annual APG round robins).

7.0 KEY WORDS

AP E-8000, aqueous, chromatography, EPA, gas chromatography, GC, QC, semi-volatiles, solids, SW-846, volatiles, water

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8.0 REFERENCES

8.1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

8.2 U.S. EPA Manual, SW-846.

9.0 REVISION HISTORY

9.1 4/10/04 – Changed SOP code from AP E-8000.

9.2 9/11/07 – Updated codes for all referenced SOPs and updated safety information.

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W-7

Sample Management Procedures

Process Owner (TS/PM/OM/LM)	
Dennis Catalano	
Assignment	Reviewed July 13, 2007 by Christine Devine
Electronic only unless colored "Controlled" stamp is present	

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1.0 SCOPE

1.1 This document specifies the duties and responsibilities of the Sample Management Office (SMO) and those of the employees coming into contact with the samples.

1.2 SMO plays a critical role in the Unit's QA program through receipt, handling, documentation, and disposal of samples for Analysis and Testing.

1.3 This SOP only applies to samples submitted to the pulp (wet) lab, paper lab, and to the analytical laboratories. Microscopy has a separate procedure for the sample management process. See MP-SAMPLE RECEIVING.

2.0 SAMPLE SUBMITTAL PROCESS

The following forms are found on the A&T website under "Forms & Templates" in the SMO folder:

- Sample Receipt Form
- Sample Analysis Request & Chain of Custody Record
- Analytical Chemistry Service Request
- Pulp & Paper Service Request
- Microstructure Service Request

To submit a sample, the following forms are found on the A&T website under "Submit a Sample" to request the sample testing schedules:

- Analytical Chemistry Service Request Form
- Pulp & Paper Service Request
- Microstructure Service Request

See section 4.0 below for more information regarding how to locate the necessary forms.

3.0 REAGENTS REQUIRED

3.1 HNO_3 (1+1) with water. Add 250 mL of conc. acid to 250 mL of water and mix. Need not be extremely accurate.

NOTE: For safety and purity reasons, this should be made up by metals laboratory personnel, not SMO personnel.

Nitric acid (concentrated), HNO_3, CAS# 7697-37-2

DANGER: Handle with extreme care. Wear safety glasses, lab coat, vinyl gloves and use in a hood. Conc. nitric acid is a strong, corrosive acid and liquid and vapor can cause severe burns. Carefully add acid to water with frequent or continuous stirring. Mix in a tub or stoppered sink. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.

- Harmful if inhaled and may cause delayed lung injury. If inhaled, can cause severe irritation or burns of mucous membranes and respiratory system, resulting in coughing, difficult breathing, chest pains, pneumonia, pulmonary edema, lung inflammation, unconsciousness, and may be fatal. Can cause severe ulceration.
- Skin contact can cause severe irritation, redness, pain, and severe skin burns. Concentrated acid causes deep ulcers and stain skin a yellow or yellow-brown color.
- Eye contact can cause severe irritation or burns and result in permanent damage.
- Nitric acid is a strong oxidizer. Contact with combustible materials, flammable materials - such as wood and solvents - or powdered metals can cause fire or explosion. Reacts with most metals to produce hydrogen gas, which can form an explosive mixture with air. Keep away from strong bases, carbides, carbonates, charcoal, (hydrogen) sulfides, cyanides, combustible materials, combustible organics or organic materials, turpentine, strong reducing agents, most common metals, carbides, ammonium hydroxide, water, and alcohols.
- Certain mixtures with benzene, 1,2-dichloroethane, or dichloromethane may be detonatable.
- Reaction products include highly toxic and dangerous fumes of reddish oxides of nitrogen having the equivalent affects as those noted above.
- Long-term exposure to concentrated vapors may cause erosion of teeth.
- Short-term exposure limit (STEL) is 10 mg/m^3 (4 ppm) and permissible exposure limit (PEL) is 5 mg/m^3 (2 ppm.)

4.0 PROCEDURE

SMO is responsible for the general cleanliness of the sample storage and receiving areas. A clean and orderly storage area is necessary for the integrity of the samples. Important records can be lost if paperwork is not kept organized.

4.1 Location of forms to submit a sample

Go to A&T web site

Click on "Submit a form". Under Service Request forms, choose from the following: Pulp and Paper, Microstructure, Sample Analysis & Testing Request with a chain of Custody form or Sample Analysis & Testing Request without a chain of custody.

4.2 Receiving Samples

Sample Management Office (SMO) receives samples through a variety of methods.

4.2.1 Samples are brought directly into SMO by the client. If no one is present in SMO, the client should consult with one of the Project Managers (PM) about his sample request.

4.2.2 Samples are shipped and/or mailed to the Technology Center via various methods.

a. Samples are accepted by our receiving personnel and delivered to the SMO. (At times, SMO will come down and retrieve samples from the receiving dock.)
b. On occasion, samples will be dropped off in the south lobby area. The receptionist will then contact SMO to pick-up the samples.

NOTE: Although section 4.1.2.b is still listed as a viable method for sample drop off, it is strongly discouraged due to safety reasons.

4.2.3 Samples are brought in to SMO by the analyst.

a. Samples are sent or given directly to the analyst by the client.

b. On occasion, samples are created by an analyst in collaboration with a client.

4.2.4 Samples are delivered to the Technology Center during off-hours.

Security handles samples as per their SOP.

a. Samples received in coolers are placed in the basement cold storage G01/G02 and security sends a voice mail message to the SMO personnel.

b. Samples not received in coolers (not to be kept chilled) are placed in the receiving area and delivered to SMO at the start of the following business day.

4.3 Project Manager Role

When samples arrive, the SMO staff calls a Project Manager (PM). The PM is determined by the client/job type received.

4.4 Unpack and evaluate the samples.

The following is a list of procedures performed in the sample check-in process. See AQ S-SampleSplitting if needed. (Section 4.4.1.j)

4.4.1 Sample handling.

THE INTEGRITY OF ALL SAMPLES SHALL BE MAINTAINED WHILE IN LABORATORY CUSTODY. FOR EXAMPLE, IF SAMPLES REQUIRE 4°C, THEY WILL BE MAINTAINED AT 4°C DURING ANY HANDLING PROCESSES (except when warming them for sub-sampling).

SAMPLES WILL BE APPROPRIATELY PROTECTED FROM CROSS-CONTAMINATION. FOR EXAMPLE, THE VOAs OR HI-RES SAMPLES WILL BE ISOLATED TO KEEP FROM POTENTIALLY CONTAMINATING OTHER SAMPLES.

a. For samples received chilled or in coolers, the SMO staff uses the Sample Receipt Form found on the A&T website under “Forms & Templates” in the SMO folder. This form is used to record, date and time samples are received, pH conditions, temperature of cooler/temperature blank and custody seal conditions, etc.

b. When temperatures are in question (i.e. no ice present or blue ice totally melted), a probe is used to check temperatures. The probes are stored in the cabinet under sample fume hood 216-4.

If there are no coolant blanks, place the probe in the cooler next to a sample bottles for the most accurate ambient temperature. Make a note on the sample receipt form how the temperatures were measured (coolant blank, water, ambient air).

c. Unpack samples and check for breakage, missing samples and correct identification, etc.

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NOTE: TOTALLY UNKNOWN SAMPLES, OR SAMPLES KNOWN OR SUSPECTED TO BE HAZARDOUS, ARE TO BE OPENED AND INSPECTED IN A LABORATORY HOOD ONLY.

Broken sample containers, irregular preservation and improper paperwork are noted on a nonconformance form as per OQ CORRACT. The assigned PM is contacted so a course of action can be taken such as the following:

- Contacting clients so more samples can be submitted
- Reducing the number of tests requested by the clients
- Contacting clients regarding future packing of samples

If anything is unclear, special or requires immediate handling about a client's request, the SMO personnel will contact the assigned PM or the appropriate analysts for assistance.

d. The SMO personnel (who unload coolers, boxes, etc.) will verify that samples and accompanying paperwork match. The SMO personnel will promptly sign all of the chain of custody forms.

When there is no client paperwork, the SMO personnel contact the project managers to see if they have any knowledge of the work that was received. When project managers have no knowledge of samples received, the SMO personnel will then look on the package for sender information (i.e. mill, company or name on the package) so that the SMO personnel or project managers can contact the sender to find out what kind of analyses are required.

All paperwork that accompanies the incoming samples is kept together and becomes part of the original SR package.

e. Determine if the samples received are within holding times.

Holding Time	Action
No Issues	Process normally
Exceeded	Contact PM, laboratories affected and complete a nonconformance f
Short	Contact PM and laboratories affected immediately

f. Determine if the samples are properly preserved.

1. Using the Sample Receipt form, confirm which samples have a pH preservation requirement and require a pH measurement to be taken. Retrieve the pH test strips (these are located in both hoods ((216-3 & 216-4)) in the SMO area.

i. To take a pH of a sample, pour a small amount of the sample into a disposable 50-mL polypropylene beaker. Dip the pH strip into the liquid.

ii. Pour excess sample down the sink with the water running and the dip sticks in the trash.

2. Most samples needing their pH checked should be preserved to a pH < 2. Samples requiring more H_2SO_4, or HNO_3 (if not designated as low level metals), may be preserved by SMO personnel or taken to the lab for preservation.

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CAUTION: Be aware that an unpreserved bottle (for metals analysis) may be for silicon testing and does not require preservation. See chain of custody and/or other testing information before adding any preservatives to such sample containers.

DANGER: Handle with extreme care. Nitric acid is a strong, corrosive mineral acid. Use all appropriate safety apparel and proceed with caution. See 'DANGER' under 3.1.

3. Cyanide pH should be > 12. If the cyanide sample has not been preserved and appears to have an improper pH, take the sample to the Lab 212 SLM and have a chemist preserve it to a pH >12 by adding 2 mL of 10N NaOH per liter of sample.

4. Use lead acetate strips to check for presence of sulfide in liquid samples where cyanide is requested. If sulfide is present in the sample, it will be indicated by a darkening of the test strip or a dark precipitate on the test strip due to PbS (lead sulfide) precipitating out with color (black). Record pH and sulfide information on Sample Receipt Form (Section 4.4.1.a).

5. Check other preservation required as specified, such as RCl_2 (residual chlorine), as necessary for N. Carolina regulatory samples.

g. Add any needed preservative in accordance with proper analytical protocol as stated in 40 CFR 136.3, Table II. See a copy of Table II in the appendix of this document.

h. Preservation is done immediately upon receipt in the lab and recorded on Sample Receipt Form.

i. If initial attempts to add preservative do not achieve the desired effect, contact an analyst to see about adding a more concentrated preservative. Total volume of preservative should not dilute the sample by more than 1 % (10 mL/L).

j. Evaluate whether there is sufficient sample to run the requested tests. If there is not, or if there is doubt, consult AQ S-SampleSplitting and/or contact the PM. Should testing proceed when there is insufficient sample, a nonconformance memo will be filled out.

k. If the samples are in an unusual or abnormal state, such as lack of proper preservative for testing (i.e. H_2SO_4 rather than HNO_3), the PM is notified and a determination is made on how to proceed. This may require a note on the sample log-in sheet under "*Comments"* or a nonconformance form may need to be completed.

4.4.2 Determine if paperwork matches samples. If not, then the PM is informed and a nonconformance memo will be filled out.

4.4.3 Evaluating Service Request (SR) needs:

a. Determine if an 'upcoming request' was created. Look in the "UPCOMING" notebook for copy.

If:	
Yes	Contact appropriate PM that samples have arrived. Proceed with log-in

	following detail on upcoming.
No	Contact appropriate PM for analysis details. These details would include: •who the client is •client location and phone number •correct project number for charging •correct tests and corresponding test codes •special instructions regarding these samples •reference SR number •confirmation of a committed turnaround time •lab doing the work

b. When there is an upcoming notice for a particular job and the samples that are received do not match the initial notice (i.e., expect 41 samples, receive only 7 in current shipment), make a copy of the upcoming notice. This copy goes with the new SR being created. Write on the original copy of the upcoming notice the number of the SR created for the work received and place the upcoming notice back into the notebook for future samples to be received for that same job.

c. Determine if there is a reference Service Request (SR).

1. A reference SR number is noted on the upcoming notice by the PM if we've done previous work for that client/project.

2. If a referenced SR number is not noted on the upcoming notice, open 'A&T SR Search' from the main screen of Laboratory Information System (LIMS).

3. A LIMS search is based on several criteria.
   i. Mill site, such as "Columbus".
   ii. Client, such as "Mike White".

4. The search is much quicker using only the upper part of the LIMS page. (Most searches will give the information required when using the upper part of the page.) Entering a test or test schedule (shown on the lower half of the page) will be much broader and take longer.

4.4.4 When the list of SRs comes up on the LIMS screen, the most recent SR is at the top (this is not true when searching the committed database). If the information being searched for is not found in the active database, try searching under the committed database. Active/committed SR's can be opened and printed by clicking on the database header in LIMS & toggling between 'Toggle act/com'.

NOTE: SRs are moved into the committed database after an SR has been closed for 120 days.

4.5 Enter sample/requested information/documentation into LIMS

After all questions are answered and issues are resolved, the SMO personnel are ready to enter the information into the Laboratory Information Management System (LIMS) and create a unique Service Request (SR) for that particular sample set. This document (SOP) does not cover the detail of sample entry into the LIMS. See LIMS notebook located in the SMO area for LIMS entry instruction.

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NOTE: SR numbers are assigned in sequential order by the computer. The SR number is a 6 digit number, with the first 2 digits designating the year, e.g., 2007 becomes 07-, and the last 4 for the sequential SR in that year, allowing up to 9999 SR's in a given year. The 4985th SR of 2007 would be 07-4985. See section 4.5.2 for labeling information.

If a sample is deleted, the LIMS does not allow that particular ID number to be reused. Not all sample ID's will be sequential.

4.5.1 After all information is entered into the LIMS and the SR is complete, print one full SR copy. Print one single copy of the front page of the SR and mark F in upper right corner. The F copy is placed in the SR file at the back of the SR and accompanying paperwork for archival filing after reporting. Scan the client paperwork into LIMS. Place all paperwork for SR in the appropriate folder.

Folder Color	Folder Contents
Green	'Permit' in title
Red	"outside" SRs
Yellow	Analytical

NOTE: No folder is made for the Cellulose Properties samples. All work is scanned into an electronic copy. However, if the Cellulose Properties SR has one or more tests for Analytical Chemistry, a yellow folder will be made and stored as in 4.5.1

Place file folder in rack next to SR printer. Notify appropriate PM electronically when SR's are ready for reviewing.

4.5.2 The PM will come to SMO and pick up SR folders. The PM will review the SR's either in SMO or at their desk to evaluate whether all aspects of the SR are in agreement with both the laboratory and the client. If no changes are required, then the PM 'releases' the SR to the laboratories (with scanned attachments) via a shared e-mail file. This puts the SR in the LIMS system so the laboratories are able to enter their data.

4.5.3 IR LAB: SMO will print a full copy of the SR, scan the client paperwork and create a folder. This folder is reviewed by a SMO technician who did not enter the SR. After review, corrections or changes, the SR is sent electronically to the IR specialist for review and released to the labs. The SR folder is placed behind the purple folder labeled Mary Beth at the back of the SR rack which is located next to the scanner. The IR specialist will either pick up the SR folder to place in the SR file cabinet drawer or SMO personnel will take it out to the drawer.

4.5.4 When the PM 'releases' the SR, it is seen in the exchange file. The SR number shows up followed by numbers. Each number represents an area of the lab. Some areas are broken down further by specialty and these show up with a letter, i.e. 4p indicates group 4 or Conventionals and the pulp area of that lab. The list below shows the number of each lab and it is the lab number that is used when sending the SR electronically. When a group opens the exchange file, they need only look for SRs with their own group number attached. Remember that all attachments are with the lab copy.

0 Project Management/QC
1 Chromatography
2 IR/Voa/Dioxin, with i (IR), v (VOA) or d (dioxin)
3 Elemental
4 Conventional, with *w* (waters) or *p (pulp)*
6 Physical Test Lab (Paper Lab)
7 Pulp Lab (Wet Lab)
8 Containerboard Lab
9 Microscopy

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Changes can be made and/or samples can be added to an SR until it is authorized (final closing).

NOTE: Archiving of completed SRs is a function of the Administrative personnel.

Exemptions

MICROSTRUCTURE:

SMO will print a full copy of the SR and scan the client paperwork as directed above. This SR is reviewed by a SMO technician who did not enter the SR. After review, corrections or changes, the SR is sent electronically to microstructure for review and released to the labs. The complete SR is put in WOW box (micro will print their own copies). The client paperwork goes in a purple folder labeled Ron which is at the back of the SR rack next to the SR scanner. The micro PM either prints this paperwork at his desk, while SMO retains the original paperwork until the file folder gets too full. At that time, SMO will discard the oldest sets of paperwork and continue to rotate paperwork in and out of that folder. Samples for Microstructure are usually delivered directly to that lab area. The procedure follows MP-SAMPLE RECEIVING.

CFTP (formerly PPTS):

SMO will print a full copy of the SR and scan the client paperwork as directed above. The SR is reviewed by a SMO technician who did not enter the SR. After review, corrections or changes, the SR is sent electronically to CFTP OM/PM for review and released to the labs. The complete SR is put in WOW box (CFTP will print their own copies). The client paperwork goes in a purple folder labeled Rick which is at the back of the SR rack next to the scanner. The CFTP OM/PM also prints this paperwork at his desk, while SMO retains the original paperwork until the file folder gets too full. At that time, SMO will discard the oldest sets of paperwork and continue to rotate paperwork in and out of that folder.

4.7 Label Samples

4.7.1 Each sample is assigned a unique lab code number. This is done through the process of logging the samples into the computer on a given SR. The lab code numbers are assigned sequentially by the computer. When new samples are "created" (such as by combining samples together), lab code numbers will be created for these as well. An SR can contain up to 999 samples.

NOTE: Generally, for work internal to the company, containers known to hold split samples from one identical source, are given the same number, being differentiated by bottle type/preservation.

Each sample number contains the 6 digit SR number, which is the two digit year, then a "–" (dash) followed by the next sequential SR number, another dash, and the first sample number, starting with 001. Each additional sample is assigned the next sequential number (e.g. 002,003, etc.). See NOTE in section 4.6.1 for more information.

Example: 00-1925-001, 00-1925-002, etc

4.7.3 Print sample labels. This is done through the LIMS. If available from the client, the label also contains the sample date and time.

4.7.4 Attach labels to each sample container. When affixing the sample number label, take care not to cover the sample detail information on the sample container such as sample ID,

date/time/preservative. Also take care not to damage samples with the label in cases where there is no container, such as with pulp sheets. In this case, attach the sample label by using a paper clip or stapling.

NOTE: See SMO staff for when to staple and when to use paper clips. Frequently, samples for the Wet Lab are taken directly to the Wet Lab by the client. (See section 4.8.12.)

4.7.5 All pulp samples going to the Paper Laboratories or to the 20 % room need to have sample labels paper clipped directly to the samples. DO NOT USE STAPLES. Ensure that sample label backing is intact so that nothing "sticky" adheres to the sample. For pulp samples requiring metals analysis, wear cotton gloves while handling samples, place samples in plastic bags and ensure no metal is touching the pulps. For pulp samples requiring Conventionals testing, wear cotton gloves while handling samples, place samples in plastic bag. For IR samples, place in envelopes. Do not staple, put in plastic or use paperclips.

4.8 Storing Samples

4.8.1 SMO personnel stores samples appropriately to maintain the integrity of the samples and the safety of the lab. Samples generating hazardous fumes require placement in a hood. Samples that will deteriorate are refrigerated or frozen. Light sensitive samples are kept in the dark.

4.8.2 There is one 65 m^3 (2,300 ft^3) walk-in cooler in the SMO area. Refrigerated samples go directly to this location after initial check-in. This refrigeration unit is large enough to accommodate a large number of sample containers. There is one 0.6 m^3 freezer (located in lab 227 SLM) for frozen samples. Non-perishable samples are kept in cupboards, shelves, and drawers in the SMO area.

4.8.3 After the Service Request is generated, label the samples and assign a storage location. (See section 4.7 - Label Samples.)

4.8.4 For each SR, complete a Sample Log Sheet. This sheet is placed in the Sample Log Book for samples processed through the SMO. There is one book for Analytical Chemistry and another for the Wet Lab, Paper Lab, & Microscopy labs. The SR is created by SMO for Microstructure, but samples do not usually come through the SMO. The Sample Log Sheet page is designed to track the location and status of samples that come into the lab. For Analytical Chemistry, each SR is assigned a separate page. Enter the SR number, the initials of the person handling the samples, the date you are placing samples in the location, the inclusive sample numbers, number of containers (bottles and bottle type (i.e.- 2X1L MET), plastic bag, envelope, etc.) and the location to which the samples are assigned.

4.8.5 For Wet Lab, Paper Lab, & Microstructure (when necessary), a modified sheet in a separate book is used in the SMO. One SR per line is used. In addition to the SR, enter the initials of the person who placed the samples in a given location, the inclusive sample numbers, the date samples are processed, and the location to which the samples are assigned.

4.8.6 For most of the routine work done for Analytical Chemistry, samples will be assigned to storage locations in the SMO and the laboratory personnel will check out samples from the SMO area. Preserved samples (in particular, those for metals) that can be stored at room temperature should be placed in cupboards C1 or C2. Liquors, caustics, acids, and samples other than waters that are held at room temperature are placed in C3. Secondary containment (plastic 'tubs') is used for all liquors, caustics, and acids. Care should be taken to store samples from each service request together and not to mix samples from more than one SR in the secondary containment 'tub.'

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WARNING: Do not store caustics and acids from the same SR together in the same tub. Accidental spills may react violently and burn or otherwise injure personnel.

4.8.7 Use the following codes to designate location in SMO:

216V: where 216 = SMO walk-in cooler and V = assigned shelf number.
D-#: where D = drawer and # =drawer number.
C-#-S: where C = cabinet, # = cabinet number, and S = shelf letter.
S-#: where S = shelf and # = shelf number.

4.8.8 EXEMPTION: VOA lab. Place all samples for volatiles analysis directly in the refrigerator located in the volatiles lab (223 SLM). This location is noted as the lab number then V-1 (223-V-1). Round Robin samples (usually small ampoules) are placed upright in a small beaker and stored in the VOA lab freezer (223 F-1).

4.8.9 When samples are picked up by the lab before being assigned a SMO location, they are noted as lab number followed by P/U (pick-up). If lab 212 SLM picks up samples directly from SMO the location is noted as 212 P/U which indicates that samples were picked up by someone from lab 212. This could be used by any lab location. On occasion, samples may be assigned to other locations. Apply an appropriate description, e.g., "northeast hood in 211," "Eatherton-233," or "G01-'S#.'" Use 'R' when designating refrigerators in lab rooms, e.g., R226N for the refrigerator in NLM 226, and 'F' for freezers. For large projects, samples may need to be kept in the cold room downstairs (G01 or G02).

4.8.10 Analysts shall sign samples out when taking them and sign them back in when they are returned. The log also contains a brief description of any sample modification or preparation, including any sub-sampling.

a. Employees taking samples will log the sample location (not the lab that the employee works in) to which the sample will be taken. At times, more than one lab will be working on the same sample and oral communication takes place regarding the current location of the sample. At the end of the shift, one of these employees logs the sample's location in the Sample Log Book sheet.

b. In the case of sample extractions requiring further analytical work, the extractives will be logged on the Sample Log sheet as to the location in SMO and with the appropriate comments as to the sample preparation performed. For example, a TCLP sample may be prepared in the Metals Lab. This sample will require further analysis in the Chromatography lab. The Metals personnel will return the TCLP sample to SMO, log its location and notify the Chromatography lab of the samples readiness and location. If the Metals personnel puts the samples in the Chromatography lab in order to be helpful, the Metals personnel must still log the location of the sample in the SMO Sample Log sheet.

4.8.11 Samples that are completely used during analysis, (except for O & G), or are returned to the client by analysts, should be noted on the sample log sheet by that analyst with the appropriate 'Disposal' and 'Comment' code noted at the bottom of the Sample Log sheet.

4.8.12 Samples for the Wet Lab are taken to Lab 105 in the high bay. Most are placed in the 50%- RH conditioning room, 105C. Room 105C has boxes and shelves noted for sample drop off. There is a clipboard in 105C where the SMO staff records the delivered samples. The information on the clipboard includes the SR#, the location of the samples and the date that the samples are delivered to the lab. Wet samples are stored in the lab refrigerator, located in lab 105 and are noted on the clipboard sheet.

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4.8.13 Most samples for the Paper Lab are placed by the paper lab personnel in the 20% conditioning room overnight. The 20% conditioning room is at the north end of the west corridor on the second floor. There is a clipboard in the 20 % conditioning room where the storage location is noted. The information on the clipboard includes the SR#, the shelf location, and the date samples were delivered to that room. If samples are clipped to wire, the location is wire. Samples for the Paper Lab are picked up daily by paper lab personnel and taken to the appropriate locations.

4.8.14 If the sample for Paper Lab requires optical testing (opacity, scattering, brightness) and is a light-sensitive product, such as newsprint, the sample should be placed (if not so received) in black plastic to prevent discoloring.

4.9 **Disposing of Samples**

4.9.1 Unless otherwise requested, the SMO holds samples that have been assigned to Analytical Chemistry for one month after closing an SR. Samples assigned to Wet Lab or Paper Lab are disposed of or returned to the client by the staff in those areas and never returned to the SMO, unless requested.

4.9.2 Final disposition could be sewering water samples, returning sample to the client, or passing appropriate samples to the Chemical Management unit.

4.9.3 Properly dispose of samples as per the disposal procedure in the appendix of the Chemical Hygiene Plan.

NOTE: If unsure of sample type or disposal method needed, see Chemical Hygiene Officer.

4.9.4 If samples were returned to the client, the return is entered into LIMS. Click on SR+Samples, then click on enter disposal to SR. Type in SR number that needs to be returned. Where it says "actual disposal", choose R for return, where it says "disposal date", enter the date that the sample was returned it to the client.

4.9.5 Upon completion of work, the sample log page is removed, updated as needed and filed in a separate notebook in numerical order. These records will be treated in the same manner as Archived Service Requests (ASR) for storage purposes. (See OQ DOCTRL.)

5.0 QUALITY ASSURANCE

5.1 The refrigerators and freezers are checked daily by assigned personnel from the lab per AQ O-DLYCHK. Refrigerators are kept in the range of 4 ± 2 °C and freezers at <-15 °C. If abnormal conditions are discovered, they are noted in the temperature logbook and the SMO is notified. SMO then takes appropriate action to correct the situation. This may include replacing a thermometer, notifying the project manager, calling (x4200) to initiate repairs, and/or transferring samples to other locations. Lab personnel also make notice of unusual situations in their daily use of the refrigerators.

5.2 Nonconformances include, but are not limited to:

- Broken sample containers
- Irregular preservation or lack of proper preservative
- No COC (Chain of Custody) or improper paperwork

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- Insufficient sample
- Received sample temperatures warm
- Id's don't match customer lists

6.0 KEY WORDS

OP S-SMO, OP S-SAMPSPLIT, sample disposal, Sample Receipt Form, Sample Log sheet, sample submission, sample management, sample management office, SMO, Chain of Custody (COC), Analytical Chemistry Service Request Form, Pulp & Paper Service Request Form

7.0 REVISION HISTORY

7.1 05 SEPT 2006: Complete revision and update of SMO processes.
7.2 01 FEB 2007: Updated document to reflect changes in Weyerhaeuser structure – PPTS is now known as CFTP.
7.3 28 FEB 2007: Added safety note to section 4.1.2, added information to section 4.4.1.d, changed SOP identification.
7.4 13 JUL 2007: Added references to the appropriate departmental SOPs, corrected typo and numbering errors, corrected section 4.5.1 and added sections a and b to 4.8.10. Also added section 4.8.11 and the appendix. Deleted Table of Contents.

Appendix

40 CFR136.3

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TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter No./name	Container [1]	Preservation [2, 3]	Maximum holding time [4]
Table IA—Bacteria Tests:			
1–4 Coliform, fecal and total	P, G	Cool, 4C, 0.008% $Na_2S_2O_3$ [5]	6 hours.
5 Fecal streptococci	P, G	Cool, 4C, 0.008% $Na_2S_2O_3$ [5]	6 hours.
Table IA—Aquatic Toxicity Tests:			
6–10 Toxicity, acute and chronic	P, G	Cool, 4 °C [10]	36 hours.
Table IB—Inorganic Tests:			
1. Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, G	do	Do.
4. Ammonia	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
9. Biochemical oxygen demand	P, G	Cool, 4°C	48 hours.
10. Boron	P, PFTE, or Quartz.	HNO_3 TO pH<2	6 months.
11. Bromide	P, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours.
15. Chemical oxygen demand	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
16. Chloride	P, G	None required	Do.
17. Chlorine, total residual	P, G	do	Analyze immediately.
21. Color	P, G	Cool, 4°C	48 hours.
23–24. Cyanide, total and amenable to chlorination.	P, G	Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid [5].	14 days.[6]
25. Fluoride	P	None required	28 days.
27. Hardness	P, G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months.
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately.
31, 43. Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
Metals:[7]			
18. Chromium VI [7]	P, G	Cool, 4 °C	24 hours.
35. Mercury [12]	P, G	HNO_3 to pH<2	28 days.
3, 5–8, 12,13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals except boron, chromium VI and mercury [7].	P, G	do	6 months.
38. Nitrate	P, G	Cool, 4°C	48 hours.
39. Nitrate-nitrite	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
40. Nitrite	P, G	Cool, 4°C	48 hours.
41. Oil and grease	G	Cool to 4°C, HCl or H_2SO_4 to pH<2.	28 days.
42. Organic Carbon	P, G	Cool to 4 °C HC1 or H_2SO4 or H_3PO4, to pH<2.	28 days.
44. Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours.
46. Oxygen, Dissolved Probe	G Bottle and top.	None required	Analyze immediately.
47. Winkler	do	Fix on site and store in dark	8 hours.
48. Phenols	G only	Cool, 4°C, H_2SO_4 to pH<2	28 days.
49. Phosphorus (elemental)	G	Cool, 4°C	48 hours.
50. Phosphorus, total	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
53. Residue, total	P, G	Cool, 4°C	7 days.
54. Residue, Filterable	P, G	do	7 days.
55. Residue, Nonfilterable (TSS)	P, G	do	7 days.
56. Residue, Settleable	P, G	do	48 hours.
57. Residue, volatile	P, G	do	7 days.
61. Silica	P, PFTE, or Quartz.	Cool, 4 °C	28 days.
64. Specific conductance	P, G	do	Do.
65. Sulfate	P, G	do	Do.
66. Sulfide	P, G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH>9.	7 days.
67. Sulfite	P, G	None required	Analyze immediately.
68. Surfactants	P ,G	Cool, 4°C	48 hours.
69. Temperature	P, G	None required	Analyze.
73. Turbidity	P, G	Cool, 4°C	48 hours.
Table IC—Organic Tests [8]			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons.	G, Teflon-lined septum.	Cool, 4 °C, 0.008% $Na_2S_2O_3$ [5].	14 days.

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Parameter No./name	Container[1]	Preservation[2,3]	Maximum holding time[4]
6, 57, 106. Purgeable aromatic hydrocarbons	...do	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] HCl to pH2[9].	Do.
3, 4. Acrolein and acrylonitrile	...do	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] adjust pH to 4–5[10].	Do.
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols[11].	G, Teflon-lined cap..	Cool, 4 °C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction.
7, 38. Benzidines[11]	...do	...do	7 days until extraction.[13]
14, 17, 48, 50–52. Phthalate esters[11]	...do	Cool, 4 °C	7 days until extraction; 40 days after extraction.
82–84. Nitrosamines[11,14]	...do	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] store in dark.	Do.
88–94. PCBs[11]	...do	Cool, 4 °C	Do.
54, 55, 75, 79. Nitroaromatics and Isophorone[11]	...do	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] store in dark.	Do.
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons[11].	...do	...do	Do.
15, 16, 21, 31, 87. Haloethers[11]	...do	Cool, 4 °C, 0.008% $Na_2S_2O_3$[5]	Do.
29, 35–37, 63–65, 73, 107. Chlorinated hydrocarbons[11].	...do	Cool, 4 °C	Do.
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs[11].			
aqueous: field and lab preservation.	G	Cool, 0–4 °C, pH<9, 0.008% $Na_2S_2O_3$.[5]	1 year.
Solids, mixed phase, and tissue: field preservation..	...do	Cool, <4 °C	7 days.
Solids, mixed phase, and tissue: lab preservation	...do	Freeze, <−10 °C	1 year.
Table ID—Pesticides Tests:			
1–70. Pesticides[11]	...do	Cool, 4°C, pH 5–9[15]	Do.
Table IE—Radiological Tests:			
1–5. Alpha, beta and radium	P, G	HNO_3 to pH<2	6 months.

Table II Notes

[1] Polyethylene (P) or glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic).

[2] Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

[3] When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under §136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See §136.3(e) for details. The term "analyze immediately" usually means within 15 minutes or less of sample collection.

[5] Should only be used in the presence of residual chlorine.

[6] Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[7] Samples should be filtered immediately on-site before adding preservative for dissolved metals.

[8] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[9] Sample receiving no pH adjustment must be analyzed within seven days of sampling.

[10] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

[12] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

[13] Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

[15] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

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W-8

Training Personnel to Perform Test Methods and Standard Operating Procedures, Procedure for

Process Owner (TS/PM/OM/LM)	
Christine Devine/Jill Roux	
Assignment	6-1-03 revised by Devine/Roux 10-21-05 revised by Devine/Roux Reviewed by Christine Devine on August 19, 2008
Electronic only unless colored "Controlled" stamp is present	

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1.0 SCOPE

1.1 This Standard Operating Procedure (SOP) is written to provide guidance for training laboratory personnel in instrument operations and testing methods and for documentation of that training.

1.2 A copy of this procedure is to be present for the trainer and trainee at the bench at time of training. This is necessary so that a procedure is performed in a proper and consistent manner.

1.3 This procedure is referred to in the Quality Manual, Section 4.6.4.b.

2.0 SUMMARY OF THE PROCEDURE

The purpose of this SOP is to familiarize the trainee with lab safety needs before starting any work in the laboratory. The trainee will be:

1. Familiarized with the method or procedure before starting any work in the laboratory. The trainer will demonstrate the entire procedure to the trainee.

2. The trainee will then perform the entire procedure under the direct supervision of the trainer. The training will continue until the trainee has generated valid data (for a test method) or demonstrated proficiency to the satisfaction of the trainer (for a standard operating procedure).

3. Acknowledgment of revisions and modifications to a procedure will be documented. An employee who has been qualified to perform a procedure and who has been doing so for an extended period of time will have this proficiency documented in lieu of training.

3.0 PROCEDURE

3.1 Weyerhaeuser Analytical Sevices Laboratory Chemical Hygiene Plan

Before any work can be performed in the laboratory by any employee, the employee must be familiarized with the Weyerhaeuser Analytical Sevices Laboratory Chemical Hygiene Plan.

3.1.1 The trainee must read the Weyerhaeuser Analytical Sevices Laboratory Chemical Hygiene Plan.

3.1.2 The trainee must then have a discussion with the Chemical Hygiene Officer for Analysis and Testing Services. This discussion will be general with regard to the plan and specific with regard to the area in which the trainee will be working.

3.2 The Training Form

3.2.1 All the training forms can be found by clicking on 'Forms and Templates' on the A&T Homepage on the Web.

3.2.1.1 The 'TrainRecord - Methods' form is used when an employee is learning a test method that is new to the employee.

3.2.1.2 The 'TrainRecord – Processes' form is used when an employee is learning a standard operating procedure that will not be used to generate data.

3.2.1.3 The 'TrainRecord – ProcedMod' form is used when an employee is receiving refresher training on a test method (for which the employee has been previously trained and for which the employee has already generated acceptable data) or demonstrated sufficient proficiency on a standard operating procedure.

3.2.1.4 The 'TrainRecord – PreviousQual" form is for an employee who has been performing a test method or standard operating procedure for an extended period of time (3 years or more) or who has developed the method or procedure. That employee is considered to be a 'grandfather' with respect to that method or procedure. A signed document stating that the employee had been previously qualified is sufficient to document the training. Proficiency must be documented. See 3.5.

3.3 Training an Employee who is learning a new test method or standard operating procedure

3.3.1 The trainee will read the test method and/or standard operating procedure. A discussion of the test method and/or standard operating procedure with the trainer will follow to be sure there are no questions with regard to the method or procedure and that the trainee has a good understanding regarding any hazards or dangers involved in performing the method or procedure.

3.3.2 The trainee will fill out the first two portions of the 'TrainRecord - Methods' form for a test method and/or the first two portions of the 'TrainRecord – Processes' form for a standard operating procedure.

3.3.3 The trainer will demonstrate the procedure from start to finish as the test method or standard operating procedure dictates. The trainer and trainee will sign the third portion of one of the 'TrainRecord' forms, entitled 'Demonstration'.

3.3.4 A secondary trainer will observe the trainee perform the procedure for a different SR when a secondary trainer is available, giving advice and guidance where needed. The trainer and trainee will sign the fourth portion of one of the 'TrainRecord' forms, entitled 'Observation'.

3.3.5 Training is complete for a test method upon production of acceptable Data/Results. The date of the data generation (completion) and service request number or IPAR (Initial Precision and Accuracy Record) are recorded. The technical specialist in charge of this particular test method will sign the 'TrainRecord - Methods' form after examining the data. Training is complete for a standard operating procedure when the trainer believes the necessary criteria and/or proficiency is met.

3.3.5.1 For environmental regulatory work, initial demonstration of capability, if required by the method, shall be kept as part of the training file. See Section 3.7.

3.3.5.2 For wet lab work, demonstration of capability is complete when the trainee successfully generates "Grande Prairie Control" data as it applies to refining, CSF and sheeting. A copy

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of the Grande Prairie Control data generated is attached and shall be kept as part of the training file when applicable.

3.3.5.3 For physical test work, demonstration of capability is complete when the trainee successfully generates "collaborative testing" and calibration testing data. A copy of the collaborative testing data generated is attached and shall be kept as part of the training file.

3.3.6 The form is then sent to the Quality Advisor for further examination and signature. Training records are then filed with the appropriate Quality Advisor.

3.4 Training an Employee to perform a test method or SOP after the test method or SOP has been updated

3.4.1 The degree of updating is important. The technical specialist must determine if a new training/IPAR is necessary. If a new training/IPAR is not necessary, then the revisions must be reviewed by the employee and documented in the 'Description of Changes' section of the 'TrainRecord – ProcedMod' form. The employee will sign and date the form in the appropriate place certifying that he/she has read, understood and agreed to perform the most recent version of each procedure listed.

3.4.2 If a new training/IPAR is necessary, these results must be documented.

3.4.3 The owner of the particular procedure (Technical Specialist, Project Manager, Operations Manager, Laboratory Manager or Quality Advisor as applicable) will then sign and date the form in the appropriate place.

3.5 Documenting the Qualifications of an Employee Qualified Long Ago

An employee who has been qualified to perform a procedure and who has been doing so for an extended period of time will have this proficiency documented in lieu of training on the 'TrainRecord – PreviousQual" form. The IPAR must be performed no matter how long an employee has been doing the test. While an employee might be able to do the method and have no training record, the employee must demonstarte the method can be done well.

The employee will list all procedures for which he/she is previously qualified with the procedure number, procedure name and the year of the date at which time the employee started performing this particular procedure.

3.6 Completion of Training

Training is not complete until the training records are filed with administrative personnnel. Until training is complete, the trainee will be directly and closely supervised by the trainer.

3.7 Training for Environmental Regulatory Work

Precision and accuracy must be demonstrated by the analyst before prepping, extracting or analyzing any samples.

3.7.1 A clean blank sample of the matrix must first be generated. Four lab control samples are then generated. Each sample is spiked with the surrogate solution as dictated by the method. Four lab control samples are spiked with a lab control spike solution made up of each single component parameter of interest. The blank sample as well as four lab control samples are extracted and analyzed. The blank sample and lab control samples should be carried through all stages of the sample preparation and measurement. . The blank sample and lab control samples must meet the

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precision and accuracy requirements stated in the method. If the method does not have these P&A requirements, then acceptable values will be determined by the LCS data of previously run batches of samples.

4.0 REVISION HISTORY

10/3/03: Added “when possible” to section 3.3.4. Fixed typos. 05/31/05: Modified 3.3.4 and 3.3.6.

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W-9

Analytical Procedure 3500

Process Owner (TS/PM/OM/LM) **Randy Eatherton**	
Assignment	Reviewed by Christine Devine July 6, 2003 Reviewed by Christine Devine July 13, 2007 Reviewed by Christine Devine September 11, 2007 Reviewed by Christine Devine August 15, 2008
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1.0 SCOPE AND APPLICATION

The procedures outlined below are based on ones from EPA SW-846.

1.1 The AM E-3500 Methods are procedures for quantitatively extracting nonvolatile and semivolatile organic compounds from various sample matrices for chromatography.

1.2 AM E-3580 describes a solvent dilution technique that may be used on non-aqueous nonvolatile and semivolatile organic samples prior to clean up and/or analysis by chromatography.

1.3 The AM E-5000 Methods are procedures for preparing samples containing volatile organic compounds for quantitative analysis.

1.4 Refer to the specific method of interest for further details.

2.0 SUMMARY OF PROCEDURE

2.1 3500 Methods: A sample of a known volume or mass is solvent extracted. The resultant extract is dried and then concentrated in a Kuderna-Danish apparatus.

2.2 5000 Methods: Refer to the specific method of interest.

3.0 INTERFERENCES

3.1 Samples requiring analysis for volatile organic compounds can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A field blank prepared from water and carried through sampling and subsequent storage and handling can serve as a check on such contamination.

3.2 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Refer to AP E-8000 for specific guidance on quality control procedures.

3.3 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further clean up of the sample extract may be necessary. Refer to AC G-3600 for guidance on clean-up procedures.

3.4 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

3.5 Glassware contamination resulting in analyte degradation: Soap residue on glassware may cause degradation of certain analytes. Specifically, aldrin, heptachlor, and most organophosphorous pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500-mL K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

4.0 APPARATUS AND MATERIALS

Refer to the specific method of interest for a description of the apparatus and materials needed.

5.0 REAGENTS

5.1 Refer to the specific method of interest for a description of the solvents needed.

5.2 Refer to method AP W-ORGSTD for preparation of all standards and spiking solutions.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See the introductory material to the organic Analyte Chapter, Section 4.1 of SW-846 and/or the individual methods.

7.0 PROCEDURE

The performance of this procedure will be dictated by the overall performance of the sample preparation in combination with the analytical determinative method.

7.1 Semivolatile organic sample extraction: Water, soil/sediment, sludge and waste samples requiring analysis for base/neutral and acid extractables and/or organochlorine pesticides must undergo solvent extraction prior to analysis. There are four procedures that may be used for this purpose: AC G-3510, AC G-3520, AC G-3540 and AC G-3550. The procedure that should be used on a particular sample is highly dependent upon the physical characteristics of that sample. Therefore, review these four procedures prior to choosing one in particular. Appropriate surrogate standards and, if necessary, matrix spiking solutions are added to the sample prior to extraction for all four procedures.

7.1.1. **AC G-3510**

Applicable to the extraction and concentration of water-insoluble and slightly water-soluble organics from aqueous samples. A measured volume of sample is solvent extracted using a separatory funnel. The extract is dried, concentrated, and, if necessary, exchanged into a solvent compatible with further analysis. AP E-3520 should be used if an emulsion forms between the solvent-sample phases, which can not be broken up by mechanical techniques.

7.1.2. **AC G-3520**

Applicable to the extraction and concentration of water-insoluble and slightly water-soluble organics from aqueous samples. A measured volume of sample is extracted with an organic solvent in a continuous liquid-liquid extractor. The solvent must have a density greater than that of the sample. The extract is dried, concentrated, and, if necessary, exchanged into a solvent compatible with

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further analysis. The limitations of Method 3510 concerning solvent-sample phase separation do not interfere with this procedure.

7.1.3. **AC G-3540**

Applicable for the extraction of nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes. A solid sample is mixed with anhydrous sodium sulfate, placed into an extraction thimble or between two plugs of glass wool, and extracted using an appropriate solvent in a Soxhlet extractor. The extract is dried, concentrated, and, if necessary, exchanged into a solvent compatible with further analysis.

7.1.4. **AC G-3550**

Applicable for the extraction of nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes using the technique of sonication. Two procedures are detailed depending upon the expected concentration of organics in the sample, a low-concentration procedure and a high-concentration procedure. In both procedures, a known weight of sample is mixed with anhydrous sodium sulfate and solvent extracted using sonication. The extract is dried, concentrated, and, if necessary, exchanged into a solvent compatible with further analysis.

7.1.5 **AC G-3580**

Applicable for the solvent dilution of non-aqueous waste samples. It is designed for wastes that may contain organic chemicals at a level greater than 20,000 mg/kg and that are soluble in the dilution solvent.

7.2 Volatile organic sample preparation: There are three procedures for volatile sample preparation: AC G-5030; AC G-5040; and direct injection. AC G-5030 is the most widely applicable procedure for analysis of volatile organics, while the direct injection technique may have limited applicability for aqueous matrices.

7.2.1. **AC G-5030**

This procedure describes the technique of purge-and-trap for the introduction of purgeable organics into a gas chromatograph. This procedure is applicable for use with aqueous samples directly and to solids, wastes, soils/sediments, and water-miscible liquids following appropriate preparation. An inert gas is bubbled through the sample, which will efficiently transfer the purgeable organics from the aqueous phase to the vapor phase. The vapor phase is swept through a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. Prior to application of the purge-and-trap procedure, all samples (including blanks, spikes, and duplicates) should be spiked with surrogate standards and, if required, with matrix spiking compounds.

7.2.2. **AC G-5041**

This procedure is applicable to the investigation of sorbent cartridges from volatile organic sampling train (VOST).

7.3 Sample analysis: Following preparation of a sample by one of the procedures described above, the sample is ready for further analysis. For samples requiring volatile organic analysis, application of one of the three methods described above is followed directly by gas chromatographic analysis: AM E-8011, AM E-8015, AM E-8021, or AM E-8031. Samples prepared for semivolatile analysis may, if necessary, undergo clean up (see AP E-3600) prior to application of a specific determinative method.

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8.0 QUALITY CONTROL

8.1 Refer to AI G-8000 for specific guidance on quality control procedures.

8.2 Before processing any samples, the analyst should demonstrate through the analysis of a water blank that all glassware and reagents are interference free. Each time a set of samples is processed, a method blank(s) should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.3 For GC or GC/MS analysis, the analytical system performance must be verified by analyzing quality control (QC) check samples. AI G-8000, Section 6.0, discusses in detail the process of verification. However, preparation of the QC check sample concentrate is dependent upon the method being evaluated.

8.3.1 **Volatile organic QC check samples**: QC check sample (LCS) concentrates containing each analyte of interest are spiked into water (defined as the LCS) and analyzed by purge-and-trap (AC G-5030). The concentration of each analyte in the LCS is a nominal 20 µg/L. The evaluation of system performance is discussed in detail in AI G-8000, beginning with section 6.5.

8.3.2 **Semivolatile organic QC check samples**: To evaluate the performance of the analytical method, the LCS must be handled in exactly the same manner as actual samples. Therefore, 500 µL of the LCS concentrate is spiked into each of four 1-L aliquots of water (from now on called the LCS), extracted, and then analyzed by GC. The variety of semivolatile analytes analyzed by GC is such that the concentration of the LCS concentrate is different for the different analytical techniques presented in the manual. AI G-8000 discusses in detail the procedure of verifying the detection system once the LCS has been prepared. The concentrations of the QC check sample concentrate for the various methods are as follows:

a. Method 8041 - Phenols: The QC check sample concentrate should contain each analyte at a concentration of 100 ug/mL in 2-propanol.

b. Method 8061 - Phthalate esters: The QC check sample concentrate should contain the following analytes at the following concentrations in acetone: butyl benzyl phthalate, 10 ug/mL; bis(2-ethylhexyl)phthalate, 50 ug/mL; di-n-octylphthalate, 50 ug/mL; and

c. Method 8081 - Orqanochlorine pesticides and PCBS: The QC check sample concentrate should contain each single-component analyte at the following concentrations in acetone: 4,4'-DDD, 10 ug/mL; 4,4'-DDT, 10 ug/mL; endosulfan II, 10 ug/mL; endosulfan sulfate, 10 ug/mL; and any other single-component pesticide at 2 ug/mL. If the method is only to be used to analyze PCBS, chlordane, or toxaphene, the QC check sample concentrate should contain the most representative multicomponent parameter at a concentration of 50 ug/mL in acetone.

d. Method 8091 - Nitroaromatics and Cyclic Ketones: The QC check sample concentrate should contain each analyte at the following concentrations in acetone: each dinitrotoluene at 20 ug/mL; and isophorone and nitrobenzene at 100 ug/mL.

e. Method 8100 - Polynuclear aromatic hydrocarbons: The QC check sample concentrate should contain each analyte at the following concentrations in acetonitrile: naphthalene, 100 ug/mL; acenaphthylene, 100 ug/mL; acenaphthene, 100 ug/mL; fluorene, 100 ug/mL;

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phenanthrene, 100 ug/mL; anthracene, 100 ug/mL; benzo(k)fluoranthene 5 ug/mL; and any other PAH at 10 ug/mL.

f. Method 8121 - Chlorinated hydrocarbons: The QC check sample concentrate should contain each analyte at the following concentrations in acetone: hexachloro-substituted hydrocarbons, 10 ug/mL; and any other chlorinated hydrocarbon, 100 ug/mL.

8.4 A surrogate standard (i.e., a chemically inert compound not expected to occur in an environmental sample) should be added to each sample, blank, and matrix spike sample just prior to extraction or processing. The recovery of the surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within the acceptance limits. Recommended surrogates for different analyte groups follow; however, these compounds, or others that better correspond to the analyte group, may be used for other analyte groups as well. Normally three or more standards are added for each analyte group.

8.5 A reagent blank, a matrix spike, and a duplicate or matrix spike duplicate must be performed for each analytical batch (up to a maximum of 20 samples) analyzed. The recovery of matrix spiking compounds indicates the presence or absence of unusual matrix effects.

8.6 The performance of this method will be dictated by the overall performance of the sample preparation in combination with the analytical determinative method.

9.0 KEY WORDS

Extractions, surrogates, matrix spike

10.0 REFERENCES

EPA SW-846 manual

11.0 REVISION HISTORY

11.1 4/10/04 – Changed SOP code from AP E-3500.

11.2 7/13/07 – Updated the SW-846 Methods series numbers in section 8.3.2.

11.3 9/11/07 – Updated the codes for the referenced SOPs.

11.4 8/15/08 – Updated the codes for the referenced SOPs.

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W-10

Extraction by Continuous Liquid-Liquid Extractor

Process Owner (TS/PM/OM/LM) **DevineC**	
Assignment	Reviewed April 19, 2004 by Christine Devine Reviewed August 19, 2008 by Christine Devine
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1.0 SCOPE

The following procedure describes the extraction of water samples by continuous liquid/liquid extraction using heavier than water solvents. Many semivolatile organic compounds may be extracted utilizing this method.

2.0 SUMMARY OF METHOD

Water samples are added to a continuous liquid/liquid extraction apparatus containing methylene chloride. Surrogate and spike standards are added and the pH is adjusted. The extraction is conducted for a minimum of 18 hours.

3.0 INTERFERENCES

Interferences can come from solvents, glassware, reagents, plastics (phthalate contamination), and equipment.

4.0 EQUIPMENT REQUIRED

4.1 250-mL round-bottom flask and glass stopper (2 each)

4.2 Continuous liquid-liquid extractor

4.3 Liquid-liquid extractor stand

4.4 Heating source (hot plate or mantel)

4.5 Teflon boiling chips rinsed 3 times with methylene chloride

4.6 Four-chamber condenser

4.7 1000-mL graduated cylinder

4.8 250-mL graduated cylinder

4.9 500-uL and/or 1.0-mL Hamilton gas-tight syringe

4.10 1 mL glass Pasteur pipet with latex bulbs

4.11 pH strips with range of 1-14

4.12 Holder for 250-mL round bottom flask

4.13 Stir rods

5.0 REAGENTS REQUIRED

5.1 25% $NaOH/H_2O$ (25g/100 mL water) extracted 3 times with hexane.

DANGER: Degree of hazard depends upon form (solid, liquid) or concentration. Skin contact with the solid hydroxides or concentrated solutions can cause rapid tissue destruction and severe burns. Handle with care. Wear safety goggles and a plastic apron. Sodium hydroxide is a strong, corrosive base and the solid or liquid forms can cause severe burns. Carefully add caustic to water with frequent or continuous stirring. Mix in a tub or stoppered sink. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.

Harmful if inhaled. Effects from inhalation of dust or mist (in such cases, use in a hood) vary from mild irritation to serious damage to tissue of the mucous membranes and upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Severe pneumonitis may occur.

Inhalation may result in spasm, inflammation and edema of the larynx and bronchi, chemical pneumonitis and pulmonary edema. Symptoms of exposure may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting.

Skin contact can cause severe irritation or severe burns and scarring with greater exposures.

Eye contact can cause irritation of eyes, and with greater exposures, it can cause burns that may result in permanent impairment of vision or blindness.

Very hygroscopic. Can slowly pick up moisture from air and react with carbon dioxide from air to form sodium carbonate.

Sodium hydroxide in contact with strong acids and organic halogen and chloro-organic compounds, especially trichloroethylene, may cause violent reactions. Contact with nitromethane and other similar nitro compounds causes formation of shock-sensitive salts. Contact with metals such as aluminum, magnesium, tin, and zinc cause formation of flammable/explosive hydrogen gas.

Avoid peroxides, flammable liquids, and chloroform/methanol mixtures.

Sodium hydroxide, even in fairly dilute solution, reacts readily with various sugars to produce carbon monoxide.

Permissible exposure limit (PEL) for dust is 2 mg/m^3 (Ceiling).

5.2 1+1 (v/v) H_2SO_4 (sp gr 1.84)/H_2O extracted 3 times with methylene chloride

DANGER: Degree of hazard depends upon concentration. Conc. acid mainly refers to those over 52 %.

Handle with care. Wear safety goggles and a plastic apron. Use in a hood if mist or fumes generated. Conc. sulfuric acid is a strong, corrosive acid and can cause severe burns. Very carefully add acid to water with frequent or continuous stirring. Mix in a tub or stoppered sink. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.

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Conc. sulfuric acid is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes and skin.

Inhalation of mist or heated fumes may result in spasm, inflammation and edema of the larynx and bronchi, chemical pneumonitis and pulmonary edema. Symptoms of exposure may include burning sensation or irritation of the nose and throat, coughing, wheezing, laryngitis, shortness of breath, labored breathing, headache, nausea and vomiting.

Avoid skin contact. Conc. acid is extremely corrosive. Symptoms of redness, pain, and severe burn can occur. Circulatory collapse with clammy skin, weak and rapid pulse, shallow respirations, and scanty urine may follow skin contact or ingestion. Circulatory shock is often the immediate cause of death.

Avoid eye contact. Extremely corrosive. Sulfuric acid can cause severe irritation or burns and result in permanent damage. Contact can cause blurred vision, redness, pain and severe tissue burns. Strong concentrations will probably cause blindness.

Long-term exposure may cause erosion of teeth.

Sulfuric acid is an oxidizer and strong dehydrating agent. May cause ignition of finely divided organic materials on contact. Reacts with most metals to produce hydrogen gas, which can form an explosive mixture with air. Keep away from bases, carbides, chlorates, fulminates, nitrates, picrates, cyanides, alkali halides, zinc iodide, permanganates, hydrogen peroxide, azides, perchlorates, nitromethane, phosphorous, nitrites, halogens, metal acetylides, oxides and hydrides, metals (especially finely powdered ones--yields hydrogen gas), strong oxidizing and reducing agents, water, and many other reactive substances.

Reaction products include hydrogen, from reaction with metals, or sulfur oxide fumes when heated.

Permissible exposure limit (PEL) is 1 mg/m^3.

Mist is a known carcinogen, depending on duration and level of exposure.

5.3 Methylene Chloride

DANGER: All manipulations involving methylene chloride must be performed within a fume hood or a sealed system to prevent venting into the laboratory. It is non-flammable.

Avoid breathing vapors. Exposure can cause anesthetic or narcotic effects, light-headedness, nausea, vomiting and headache. Excessive exposure may cause irritation to upper respiratory tract. Unconsciousness and death may result from extreme cases of over exposure. High levels may also cause cardiac arrhythmias (irregular heartbeats). Breathing vapors can elevate carboxyhemoglobin levels in the cardiovascular system thereby impairing the blood's ability to transport oxygen. Persons who smoke tobacco products will experience an intensified elevation of carboxyhemoglobin levels. Observations in animals include liver and kidney effects.

Delayed effects: exposure may aggravate symptoms of angina (chest pains). Liver and kidney damage may occur.

Avoid skin contact. Prolonged or repeated exposure may cause skin irritation, even a burn. Repeated contact may cause drying or flaking of skin. Can cause irritation and dermatitis.

Cancer information: Suspect carcinogen. Causes cancer in mice and benign tumors in rats.

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Avoid direct sunlight and UV sources. High temperatures and open flame may produce phosgene. Incompatibility with metals such as: aluminum powders, potassium, sodium, and zinc powder. Avoid unintended contact with amines. Avoid contact with strong bases and strong oxidizers. Avoid prolonged contact with or storage in aluminum or its alloys.

OSHA action level: 12.5 ppm. PEL is 25 ppm. (If you can smell it, the amount is too high.)

5.4 Acetone

DANGER: Acetone is highly flammable. It has a flash point of -20 °C (-4 °F) and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity).

Avoid breathing vapors. Exposure can cause coughing, dullness, headache, dizziness, nausea, irritation to the eyes and respiratory tract, narcosis, and unconsciousness. Higher concentrations may cause damage to kidneys, liver, and central nervous system (depression). Chronic exposure during pregnancy may be harmful.

Avoid skin contact. Acetone is absorbed through the skin. Prolonged or repeated skin contact can cause severe irritation and dermatitis, because of the defatting action on skin. May cause redness, pain, drying, and cracking of the skin. Toxicity of alcohol and halogentated hydrocarbons may be increased.

Acetone can react vigorously with oxidizing materials. Avoid strong acids, strong alkalais, and halogens and halogen compounds.

5.5 Hexane

DANGER: All manipulations involving hexane must be performed within a fume hood. n-Hexane is highly flammable. It has a flash point of -23 °C (-9 °F), has explosive limits in air in the range of 1 - 7 %, and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity). n-Hexane can react vigorously with oxidizing materials.

Avoid breathing vapors. Exposure can cause dizziness, numbness of extremities, and intoxication. n-Hexane is a central nervous system depressant and neurotoxin. Acute exposure causes irritation, narcosis, and gastrointestinal tract irritation. Chronic inhalation causes peripheral neuropathy can have neurotoxic effects.

Avoid skin contact. n-Hexane is absorbed through the skin. Prolonged or repeated skin contact can cause irritation and dermatitis, through defatting of skin.

5.6 Surrogate and matrix spiking solutions (See AC G-3500)

5.7 Pesticide-grade methylene chloride for trace analysis

5.8 High purity grade acetone suitable for gas chromatography

6.0 PROCEDURE

6.1 All glassware should be washed by glassware personnel in accordance with normal organic glassware cleaning procedures prior to use. See AC G-3500. Rinse the liquid-liquid extractor, 250-

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mL round bottom, 1000-mL graduated cylinder, liquid-liquid condenser and 250-mL graduated cylinder 2 times with methylene chloride and allow to drain.

6.2 Place the 250-mL round-bottom flask in the holder. Add 4-5 Teflon boiling chips and, in a hood, add 100-125mL of methylene chloride.

6.3 In a hood, add 200-250 mL methylene chloride to the liquid-liquid extractor, making sure that the level of solvent is above the glass arm protruding from the bottom of the extractor. Attach the 250-mL round bottom to the liquid-liquid extractor and set the round bottom in the heating mantle. Connect the extractor to the stand securely, making sure the extractor is vertical.

6.4 NOTE: Exposure to concentrations of methylene chloride exceeding safe levels may occur. Use caution when working with methylene chloride outside the hood, especially when adding the sample to the apparatus or taking it down. Pour slowly and carefully to minimize vapors and keep face away from the liquid/liquid extractor. Make sure solvent is cool before replacing or removing flasks from extractors.

Allow samples to come to room temperature. Vigorously shake sample and measure 500 mL into the graduated cylinder. Slowly pour the sample into the liquid-liquid extractor. Record the sample number and volume in the notebook. Add millipore water to the extractor until the methylene chloride begins to flow into the round bottom flask. NOTE: Sample volume used for extraction may be adjusted, depending upon total amount of sample, sample matrix, or desired detection limits, up to a maximum of 1 L.

6.5 Rinse a Hamilton 1.0-mL or 500-uL syringe 3 times with acetone. Draw surrogate into the syringe, pull and push the plunger a few times to rinse the syringe with the surrogate. Add appropriate amount of surrogate, as indicated on the chromatography tracking sheet, to the sample in the liquid-liquid extractor. Record the surrogate standard number and the volume added to the sample in the notebook.

6.6 If the sample is a matrix spike or matrix spike duplicate sample, rinse a 1.0-mL or 500-uL syringe with acetone and matrix spiking solution as in Step 6.5. Add the appropriate amount of the matrix spiking solution, as indicated on the chromatography tracking sheet, to each of the two matrix spike samples. Record the spike standard number and the volume added to the sample in the notebook.

6.7 Using a 9” Pasteur pipette, stir the sample, then measure the pH by wetting a strip of wide range pH paper with a drop of sample from the pipette. Using the NaOH and H_2SO_4 adjust the pH to appropriate level (stir the sample each time with clean pipette prior to measuring the pH).

6.8 Connect the liquid-liquid condenser making sure cold water flows freely through it. Add a bead of H_2O to the two joints and tighten the joints. The H_2O will prevent possible freezing of the joints and seal the joints to prevent solvent vapor loss.

6.9 Turn the heat source on so that the solvent moderately boils (drip rate of 5-15 mL/min), usually a setting of “5” on the heat controller. Let the extraction continue for a minimum of 18 hours. If EPA Method 625 is the determinative method, the minimum extraction time is 24 hours.

6.10 Turn the heat source off and allow it to cool for 1 hour. Remove the condenser, and lift the liquid-liquid extractor so that the 250-mL round-bottom flask can be removed. The extract is now ready for concentration.

6.11 If extraction of the same sample using a different pH is necessary, replace the removed flask with a clean flask that contains boiling chips and 100 – 125 mL methylene chloride. Adjust the pH to the

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appropriate level according to Section 6.7, and then proceed with extraction according to Section 6.8 – 6.10.

6.12 Once the last extraction is complete and the extract removed, the contents of the extractor must be disposed of properly.

6.13 Create a waste container by placing a yellow Hazardous Waste Identification label on an empty 4-L solvent bottle and marking out any legible portions of the original label. Fill in the sections of the hazard label and identify the contents as ≈ 80% methylene chloride and ≈ 20% water.

6.14 Remove the flask and carefully drain the remaining methylene chloride in the extractor out the arm and into the solvent waste container. Drain until all the methylene chloride has been removed and the water starts flowing into the waste container. The remaining water may be disposed down the drain with the water running. Rinse extractor 2 or 3 times with water, then place under faucet and let water fill extractor until next one is ready to rinse. Rinsed extractors may be place in glassware bins or on a cart to be taken to glasswashing. Repeat 6.14 for each extractor.

7.0 QUALITY CONTROL

Refer to the determinative method for specific quality control requirements.

8.0 KEYWORDS

liquid/liquid extractor, methylene chloride, round-bottom flask, sulfuric acid, sodium hydroxide

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W-11

Sonication for Soils and Sediments

Process Owner (TS/PM/OM/LM)	
Randy Eatherton	
Assignment	Reviewed July 13, 2007 by Christine Devine
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1.0 SCOPE AND APPLICATION

1.1 This procedure is based on EPA Method 3550, and is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes. The sonication process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 The procedure is divided into two sections, based on the expected concentration of organics in the sample. The low concentration method (individual organic components of <20 mg/kg) uses a larger sample size and a more rigorous extraction procedure (lower concentrations are more difficult to extract). The high concentration method (individual organic components of >20 mg/kg) is much simpler and therefore faster.

1.3 It is highly recommended that the extracts be cleaned up prior to analysis. See GPC Cleanup SOP, AC G-3640.

2.0 SUMMARY OF METHOD

2.1 LOW CONCENTRATION METHOD

A 30-g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent-extracted three times using sonication. The extract is separated from the sample by vacuum filtration or centrifugation. The extract is ready for cleanup and/or analysis following concentration.

2.2 HIGH CONCENTRATION METHOD

A 2-g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent-extracted once using sonication. A portion of the extract is removed for cleanup and/or analysis.

3.0 INTERFERENCES

Refer to EPA Method 3500. In addition, avoid contact of samples, equipment, and reagents with any form of plastic or rubber materials including gloves, hand lotion, pH buffers, and ink.

4.0 ESTIMATE OF PREPARATORY TIME

6-8 hours for a set of 12 low-concentration samples, 4-6 hours for a set of 12 high-concentration samples. This includes extraction, sample concentration, and GPC loading. An additional 2-3 hours is required to complete preparation after GPC clean-up. GPC cleanup takes approximately 1-2 hours per sample.

5.0 AMOUNT OF SAMPLE

Typically 30 g are used for low concentration samples and 2 g are used for high concentration samples. Aliquots may vary based on desired detection limits.

6.0 SAMPLE HANDLING AND STORAGE

6.1 Sediment/soil samples: Decant and discard any water layer on a sediment sample down the sink. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks in the trash.

6.2 Waste samples that are dry and amenable to grinding: Grind or otherwise subdivide the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. Introduce sufficient sample into the grinding apparatus to yield at least 10 g after grinding.

6.3 Finished extracts must be stored in 4°C ± 2°C refrigerator until analysis.

7.0 APPARATUS AND MATERIALS

7.1 GRINDING

If samples require grinding and the sample will not pass through a 1-mm standard sieve or cannot be extruded through a 1-mm opening, it should be processed into a homogeneous sample that meets these requirements. Fisher Mortar Model 155 Grinder, Fisher Scientific Co., Catalogue Number 8-323, or an equivalent brand and model, is recommended for sample processing. This grinder should handle most solid samples, except metallic, gummy, fibrous, or oily materials.

A Sonics Materials "Vibra Cell" horn-type sonicator equipped with a titanium tip will be used for all sonication. Minimum of 375 watts.

7.2 PERCENT MOISTURE

See test method, AM S-2540G.

7.3 VACUUM FILTER

7.3.1. 150-mL fritted Buchner funnel, coarse or medium frit.

7.3.2. 500-mL vacuum flask.

7.4 K-D

See SOP AC G-CONC.

7.5 OTHER EQUIPMENT

7.5.1. Teflon boiling chips, solvent extracted

7.5.2. Steam table, with concentric ring cover

7.5.3. Syringe: 5-mL.

7.5.4. Pasteur pipets: 23-cm (9-inch)

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7.5.5. 300-mL beakers, 400-mL beakers.

8.0 REAGENTS

All Solvents must be pesticide grade or better.

8.1 DRYING AGENT

8.1.1. Anhydrous and reagent grade, Sodium Sulfate. Purify by heating at 400°C for 4 hours. Cool in a desiccator and store in a bottle.

8.1.2. Heated at 400°C for 4 hr.

8.1.3. Cooled and stored in a desiccator.

8.2 SOLVENTS

8.2.1. Methylene chloride:acetone (1:1, v:v)

8.2.2. Methylene chloride.

8.2.3. Hexane.

9.0 PROCEDURE

9.1 DETERMINATION OF MOISTURE CONTENT

In certain cases, sample results are desired based on a dry-weight basis. When such data is desired, a portion of sample for moisture determination should be weighed out at the same time as the portion used for analytical determination. Refer to AM S-2540G.

9.2 pH DETERMINATION

Refer to test method, WM E-pH, if pH of sample is desired.

9.3 LOW CONCENTRATION EXTRACTION

9.3.1. The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh approximately 30 g of sample into a 400-mL beaker. Record the mass to the nearest 0.1 g. Non-porous or wet samples (gummy or clay type) that do not have a free-flowing sandy texture must be mixed with 60 g of anhydrous sodium sulfate using a spatula. The sample should be free-flowing at this point. Add surrogate standards to all samples, spikes, and blanks as indicated on the chromatography tracking sheet. For the sample in each analytical batch selected for spiking, add the matrix spiking standard as indicated on the chromatography tracking sheet. Immediately add 100 mL of the extraction solvent, 1:1 methylene chloride/acetone (Section 8.2.1).

9.3.2 Place the bottom surface of the tip of the #207 3/4-inch disrupter horn about 1.25 cm (1/2 inch) below the surface of the solvent, but above the sediment layer.

9.3.3. Sonicate for 3 min, with the output control knob set at 10, with the mode switch set on "Pulse" and percent-duty cycle knob set at "50%". Do NOT use a microtip probe.

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9.3.4. Decant and filter extraction solvent through a fritted Buchner funnel into a 500-mL vacuum flask using vacuum filtration.

9.3.5. Repeat the extraction two or more times with two additional 100-mL portions of extraction solvent. Decant off the extraction solvent after each sonication. On the final sonication, pour the entire sample into the 150-mL fritted Buchner funnel and quantitatively transfer with a portion of the 1:1 acetone/methylene chloride extraction solvent.

9.3.6. Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask.

9.3.7. Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Wash the extractor flask and sodium sulfate column with 100-125 mL of extraction solvent to complete the quantitative transfer.

9.3.8. Concentrate the extract as described in SOP AC G-CONC.

9.3.9. If a solvent exchange is required, (for pesticides or PCB), add a minimum of 10 mL of the exchange solvent through the top of the Snyder column and re-concentrate.

9.3.10. Concentrate or adjust the extract to the final volume indicated on the chromatography tracking sheet, as described in SOP AC G-CONC.

9.3.11. If sulfur crystals are a problem, proceed to SOP AC G-3660B for cleanup.

9.3.12. Transfer the concentrated extract to a clean screw-cap vial, taking care to rinse the concentration vessel three times with solvent in use to ensure quantitative transfer. Seal the vial with a Teflon-lined lid and mark the level on the vial. Label with the sample number and fraction and store in the dark at 4°C until ready for analysis or cleanup.

9.4 HIGH CONCENTRATION EXTRACTION

NOTE: Amount of sample, standards, and solvent may be doubled to ensure adequate volume of extract for GPC clean-up.

9.4.1. Transfer approximately 2 ±0.1g of sample to a tared 20-mL vial. Wipe the mouth of the vial with a tissue to remove any sample material. Record the exact weight of sample taken. Cap the vial before proceeding with the next sample to avoid any cross contamination.

9.4.2. Add 2 g of anhydrous sodium sulfate to sample in the 20-mL vial and mix well.

9.4.3. Surrogate standards are added to all samples, spikes, and blanks. Add the surrogate spiking solution to sample mixture as indicated on the chromatography tracking sheet. For the sample in each analytical batch selected for spiking, add the matrix spiking standard as indicated on the chromatography tracking sheet.

9.4.4. Immediately add a volume of methylene chloride necessary to bring the final volume of 10.0 mL considering the added volume of surrogates and matrix spikes. Disrupt the sample with the 32-mm (1/8-in.) tapered microtip ultrasonic probe for 2 min at output control setting 5 and with mode switch on continuous and percent duty cycle of 50%.

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9.4.5. Loosely pack disposable Pasteur pipets with 2- to 3-cm Pyrex glass-wool plugs. Filter the extract through the glass wool and collect 5.0 mL in a concentrator tube if further concentration is required. Concentrate the 5.0-mL extract to 1.0 mL unless instructed differently.

9.4.6. The extract is ready for cleanup or analysis, depending on the extent of interfering co-extractives.

10.0 QUALITY CONTROL

Any reagent blanks, matrix spikes, or control samples should be subject to exactly the same analytical procedures as those used on actual samples.

11.0 REFERENCES

11.1 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

11.2 U.S. EPA, Interlaboratory Comparison Study: Methods for Volatile and Semi-Volatile Compounds, Environmental Monitoring Systems Laboratory, Office of Research and Development, Las Vegas, NV, EPA 600/4-84-027, 1984

12.0 KEYWORDS

nonvolatile, semivolatile organic compounds, sonication, soil, sludges, wastes, sediments, clean-up

13.0 REVISION HISTORY

13.1 7/13/07 – Updated SOP and test method codes as these documents have been updated.

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W-12

Acid Digestion of Aqueous Samples & Extracts for Metals Analysis by ICP and ICPMS

Process Owner (TS/PM/OM/LM)	
Dan Deprez	
Assignment	Reviewed April 25, 2006 by Christine Devine Reviewed March 6, 2007 by Christine Devine

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1.0 SCOPE AND APPLICATION

1.1 This method is equivalent to EPA SW-846 method 3010 and EPA-600/4-79-020 section 4.1.3 in the metals portion. This digestion procedure is used for the preparation of aqueous samples, TCLP extracts, and wastes that contain suspended solids for analysis by inductively coupled argon plasma spectroscopy (ICP) and inductively coupled argon plasma mass spectroscopy (ICPMS).

1.2 This digestion method is used for dissolved metals if digestion is required. Dissolved metals do not require digestion if the sample meets the following criteria:

1.2.1 Has a COD of <20;

1.2.2 Is visibly transparent with a turbidity of 1 NTU or less;

1.2.3 Is colorless with no perceptible odor;

1.2.4 Is of one liquid phase;

1.2.5 Is free of particulate or suspended matter following acidification.

1.3 Elements typically analyzed from this method are:

Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Sn, Sr, Ti, Tl, V, Zn

1.4 This method replaces AM 1-3010.

2.0 SUMMARY OF METHOD

A representative sample is digested with nitric acid and refluxed with hydrochloric acid.

3.0 INTERFERENCES

Interferences are discussed in the analytical SOP for each instrument.

4.0 ESTIMATE OF ANALYTICAL TIME

This method should take 12 - 16 hours.

5.0 AMOUNT OF SAMPLE REQUIRED

A minimum of 50 mL of sample is required which may not allow for QC. A more desirable amount would be 500 mL - 1000 mL to allow for other analyses.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Samples should be preserved to a pH of < 2 with HNO_3 or refrigerated at a temperature of 4 °C. Unknown samples should be considered hazardous. Vinyl gloves should be worn when handling samples. If a sample produces a noxious odor, try to work with the sample in a hood. Prepared samples (digestates) will be stored for a minimum of 1 month and then dumped into the disposal drum. When the disposal drum is full it will be transferred to the chemical management unit for proper disposal.

7.0 REAGENTS

Reagents must not contain detectable analytes of interest.

7.1 Nitric acid (concentrated), HNO_3. CAS # 7697-37-2

DANGER: Handle with care. Wear safety glasses, vinyl gloves, lab coat, and use in a hood. Conc. nitric acid is a strong, corrosive acid and liquid and vapor can cause severe burns. Carefully add acid to water with frequent or continuous stirring. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.
Harmful if inhaled and may cause delayed lung injury. If inhaled, can cause severe irritation or burns of mucous membranes and respiratory system, resulting in coughing, difficult breathing, chest pains, pneumonia, pulmonary edema, lung inflammation, unconsciousness, and may be fatal. Can cause severe ulceration.
Skin contact can cause severe irritation, redness, pain, and severe skin burns. Concentrated acid causes deep ulcers and stains skin a yellow or yellow-brown color.
Eye contact can cause severe irritation or burns and result in permanent damage.
Long-term exposure to concentrated vapors may cause erosion of teeth.
Nitric acid is a strong oxidizer. Contact with combustible materials, flammable materials -such as wood and solvents - or powdered metals can cause fire or explosion. Reacts with most metals to produce hydrogen gas, which can form an explosive mixture with air. Keep away from strong bases, carbides, carbonates, charcoal, (hydrogen) sulfides, cyanides, combustible materials, combustible organics or organic materials, turpentine, strong reducing agents, most common metals, carbides, ammonium hydroxide, water, and alcohols.
Certain mixtures with benzene, 1,2-dichloroethane, or dichloromethane may be detonatable.
Reaction products include highly toxic and dangerous fumes of reddish oxides of nitrogen having the equivalent affects as those noted above.
Short-term exposure limit (STEL) is 10 mg/m^3 (4 ppm) and permissible exposure limit (PEL) is 5 mg/m^3 (2 ppm).

7.2 Hydrochloric acid (concentrated), HCl. CAS # 7647-01-0

WARNING: Handle with care. Wear safety glasses, vinyl gloves, lab coat, and use in a hood. Conc. hydrochloric acid is a strong, corrosive acid and liquid and vapor can cause burns. Carefully add acid to water with frequent or continuous stirring. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.
Avoid breathing vapors. Exposure can cause spasm, destruction to tissue of the mucous membranes, inflammation and edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Symptoms of exposure may include burning sensation, coughing, choking, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. In severe cases, inhalation may result in circulatory failure, and death.
Avoid skin contact. Hydrochloric acid can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and discolor skin.

Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Long-term exposure to concentrated vapors may cause erosion of teeth.
Highly reactive with strong bases, metals, metal oxides and alkali metals, hydroxides, amines, carbonates and other alkaline materials. Avoid contact with materials such as cyanides, sulfides, sulfites, and formaldehyde.
Mixing with strong acids may produce toxic products. Conc. sulfuric acid will dehydrate hydrochloric acid, releasing hydrogen chloride gas. Certain mixtures with nitric acid will give *aqua regia*, which is unstable and extremely corrosive, and gives off toxic gas as it decomposes.
OSHA PEL is 7 mg/m^3 (5 ppm)

8.0 PROCEDURE

SAFETY: Wear safety glasses, vinyl gloves, lab coat, and work in a hood. All heating of samples will be done in a hood.

NOTE: Sample volumes will be measured using graduated labware such that the bottom of the meniscus is at the mark for the desired amount.

NOTE: Acids and reagents will be added using a dispenser set to the desired amount.

NOTE: Sample aliquots and final volumes may vary based upon quantity of sample provided, type of sample, and client needs. The amount of acids and reagents added will be adjusted accordingly to provide the same acid concentration in the final volume.

8.1 Label hotblock tubes with the sample I.D. using a blue marker or with an "I" if using preprinted labels.

8.2 Transfer a 50-mL representative aliquot of the well-mixed sample to the appropriate labeled hotblock tube using the graduations on the side of the tube to measure.

8.3 Add 1.5 mL of conc HNO_3.

8.4 Place in a hotblock set at 95 °C and evaporate to 2 - 4 mL, except for TCLPs which will not be taken lower than 5 mL. Make certain that the samples do not boil or go dry. Remove samples from the hotblock and allow them to cool.

NOTE: If a sample goes dry, low recoveries will result. Should this occur, discard the sample and start over with a new aliquot.

8.5 Add another 1.5-mL of conc HNO_3. Cover with a watchglass and reflux for 30 - 45 min. If necessary, continue heating and adding additional conc HNO_3 in 1.5-mL portions until the digestion is complete (indicated when the digestate is light in color or does not change in appearance with continued refluxing).

8.6 If needed, remove the watchglass and evaporate to 2 - 4 mL. Remove samples from the hotblock and allow them to cool.

8.7 Add 2.5 mL conc HCl and 2.5 mL DDI-water and reflux, with watchglass, for 15 min. Remove samples from the hotblock and allow them to cool.

8.8 Dilute to a 50-mL final volume with DDI-water, cap, and mix by inverting the tubes 4 - 5 times. Allow the particulates to settle out before analysis, generally overnight. The digestate has an approximate acid concentration of 2 % HNO_3 and 5 % HCl. The sample is now ready for analysis.

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9.0 QUALITY CONTROL

9.1 A reagent blank must be processed with each analytical batch to detect contamination. The concentration of analyte in the blank should be less than the quantitation limit (QL) for each element.

9.2 A laboratory control sample, currently LCSW (Laboratory Control Sample Water = a spiked blank), must be processed with each analytical batch. The results for each reported element must be within 85 - 115 % of the true value.

9.3 A duplicate sample should be processed with each analytical batch to indicate the reproducibility of a sample.

9.4 A spiked sample should be processed with each analytical batch to determine sample matrix effects. The spike is added after the sample is aliquoted and before any acid is added.

For TCLP extracts, the spike is added after filtration and before preservation of the extract. A spike must be done with each sample type within each batch.

9.5 For NPDES samples, 10 % QC (duplicates and spikes) is required rather than 5 %.

9.6 QC results should be within the respective established QC limits.

10.0 PRECISION AND ACCURACY

10.1 Precision and accuracy are sample dependent and may vary as the sample matrix varies.

10.2 Practical quantitation limits (PQLs) are determined from the instrument detection limits (IDLs) and/or method detection limits (MDLs) pertaining to the specific instrument.

11.0 KEY WORDS

3010, AM E-3010, AM 1-3010, aqueous samples, digestion, dissolved metals, EPA, extracts, FAAS, ICP, metals, SW-846, TCLP, total, total metals, water

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste (SW-846), USEPA; Office of Solid Waste, Washington D.C.; Revised Dec, 1996.

12.2 Methods for Chemical Analysis of Water and Wastes (EPA-600/4-79-020), Environmental Monitoring and Support Laboratory, Cincinnati Ohio, Revised March 1983.

13.0 REVISION HISTORY

13.1 The method was updated on 04/25/06 to update the title, remove FAAS (now using ICPMS), update the estimate of analytical time, remove instrument PQLs (varies with prep and instrument), remove reporting and calculations (part of analysis, not the prep), change LCSW range to 85 - 115 % and no longer charting (as allowed per EPA method 200.7, 200.8, and 200.9), reword some sections, and add a note to section 8.0 (adjusting aliquots and final volumes).

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13.2 The method was updated on 03/02/07 to include additional safety information and to reword some sections.

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W-13

Acid Digestion of Sediments, Soils, Sludges, and other solid samples for analysis by ICP and ICPMS

Process Owner (TS/PM/OM/LM) **Dan Deprez**	
Assignment	Reviewed September 9, 2004 by Christine Devine Reviewed April 25, 2006 by Christine Devine Reviewed October 5, 2006 by Christine Devine Reviewed March 6, 2007 by Christine Devine
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Proprietary — Disclosure limited to persons confidentially bound to Weyerhaeuser.

1.0 SCOPE AND APPLICATION

1.1 This method is equivalent to EPA SW-846 method 3050. This method is an acid digestion procedure used to prepare sediments, soils, sludges, and other solid samples for analysis by inductively coupled argon plasma spectroscopy (ICP) and inductively coupled argon plasma mass spectroscopy (ICPMS).

1.2 Elements typically analyzed from this method are:

Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Sn, Sr, Ti, Tl, V, Zn

1.3 This method replaces AM 1-3050.

2.0 SUMMARY OF METHOD

A representative sample is digested in nitric acid and hydrogen peroxide and refluxed with hydrochloric acid.

3.0 INTERFERENCES

Interferences are discussed in the analytical SOP for each instrument.

4.0 ESTIMATE OF ANALYTICAL TIME

This procedure should take 8 - 16 hours.

5.0 AMOUNT OF SAMPLE REQUIRED

A minimum of 5 grams of solid material is required which may not allow for QC. A more desirable amount would be 50 grams of solid material to allow for other analyses.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Samples should be refrigerated at a temperature of 4 °C. Unknown samples should be considered hazardous. Vinyl gloves should be worn when handling samples. If a sample produces a noxious odor, try to work with the sample in a hood. Prepared samples (digestates) will be stored for a minimum of 1 month and then dumped into the disposal drum. When the disposal drum is full it will be transferred to the chemical management unit for proper disposal.

7.0 REAGENTS

Reagents must not contain detectable analytes of interest.

7.1 Nitric acid (concentrated), HNO_3. CAS # 7697-37-2

DANGER: Handle with care. Wear safety glasses, vinyl gloves, lab coat, and use in a hood. Conc. nitric acid is a strong, corrosive acid and liquid and vapor can cause severe burns. Carefully add acid to water with frequent or continuous stirring. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.
Harmful if inhaled and may cause delayed lung injury. If inhaled, can cause severe irritation or burns of mucous membranes and respiratory system, resulting in coughing, difficult breathing, chest pains, pneumonia, pulmonary edema, lung inflammation, unconsciousness, and may be fatal. Can cause severe ulceration.
Skin contact can cause severe irritation, redness, pain, and severe skin burns. Concentrated acid causes deep ulcers and stains skin a yellow or yellow-brown color.
Eye contact can cause severe irritation or burns and result in permanent damage.
Long-term exposure to concentrated vapors may cause erosion of teeth.
Nitric acid is a strong oxidizer. Contact with combustible materials, flammable materials -such as wood and solvents - or powdered metals can cause fire or explosion. Reacts with most metals to produce hydrogen gas, which can form an explosive mixture with air. Keep away from strong bases, carbides, carbonates, charcoal, (hydrogen) sulfides, cyanides, combustible materials, combustible organics or organic materials, turpentine, strong reducing agents, most common metals, carbides, ammonium hydroxide, water, and alcohols.
Certain mixtures with benzene, 1,2-dichloroethane, or dichloromethane may be detonatable.
Reaction products include highly toxic and dangerous fumes of reddish oxides of nitrogen having the equivalent affects as those noted above.
Short-term exposure limit (STEL) is 10 mg/m^3 (4 ppm) and permissible exposure limit (PEL) is 5 mg/m^3 (2 ppm).

7.2 Hydrochloric acid (concentrated), HCl. CAS # 7647-01-0

WARNING: Handle with care. Wear safety glasses, vinyl gloves, lab coat, and use in a hood. Conc. hydrochloric acid is a strong, corrosive acid and liquid and vapor can cause burns. Carefully add acid to water with frequent or continuous stirring. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.
Avoid breathing vapors. Exposure can cause spasm, destruction to tissue of the mucous membranes, inflammation and edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Symptoms of exposure may include burning sensation, coughing, choking, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. In severe cases, inhalation may result in circulatory failure, and death.
Avoid skin contact. Hydrochloric acid can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and discolor skin.
Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Long-term exposure to concentrated vapors may cause erosion of teeth.
Highly reactive with strong bases, metals, metal oxides and alkali metals, hydroxides, amines, carbonates and other alkaline materials. Avoid contact with materials such as cyanides, sulfides, sulfites, and formaldehyde.
Mixing with strong acids may produce toxic products. Conc. sulfuric acid will dehydrate hydrochloric acid, releasing hydrogen chloride gas. Certain mixtures with nitric acid will give *aqua regia*, which is unstable and extremely corrosive, and gives off toxic gas as it decomposes.
OSHA PEL is 7 mg/m^3 (5 ppm)

7.3 Hydrogen peroxide (30 %) H_2O_2. CAS # 7722-84-1

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DANGER: Handle with care. Wear safety glasses, vinyl gloves, lab coat, and use in a hood.
Hydrogen peroxide is extremely destructive (causes burns) to tissue of the mucous membranes and upper respiratory tract, eyes and skin.
Avoid inhalation (from mists). May result in spasm, inflammation and edema of the larynx and bronchi, chemical pneumonitis, pulmonary edema, and lead to frequent attacks of bronchial infection. Symptoms of exposure may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. It is a lung sensitizer.
Avoid eye contact: May cause burns, damaging the lens or cornea.
Hydrogen peroxide is a possible cancer hazard. It is a proven carcinogen for in animal studies.
OSHA: TWA: 1.4 mg/m^3, 1 ppm.
Hydrogen is a strong oxidizer and corrosive. Contact with combustible material may cause fire or explosion.
Incompatibilities: sensitive to light, rust, brass, iron and iron salts, finely powdered metals, copper, copper alloys, galvanized iron, zinc, and nickel.
Storage: keep container in a cool (refrigeration), well-ventilated area, away from light. Separate from acids, alkalies, reducing agents and combustibles. May develop pressure and burst container.

8.0 PROCEDURE

SAFETY: Wear safety glasses, vinyl gloves, lab coat, and work in a hood. All heating of samples will be done in a hood.

NOTE: Sample volumes will be measured using graduated labware such that the bottom of the meniscus is at the mark for the desired amount.

NOTE: Acids and reagents will be added using a dispenser set to the desired amount.

NOTE: Sample aliquot and final volumes may vary based upon quantity of sample provided, type of sample, and client needs. The amount of acids and reagents added will be adjusted to provide the same acid concentration in the final volume.

8.1 Label hotblock tubes with the sample I.D. using a blue marker or with an "I" if using preprinted labels.

8.2 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh, to the nearest 0.001 g, the following amount of sample into the appropriate labeled hotblock tube:

sample type	aliquot in grams	additional instructions
soil and solid samples	0.5 g	For “wet” samples, use 0.5 g equivalent solids (up to 5 g of sample) to maintain the QLs. For RCRA, use as-received samples.
oils	0.5 g	Wait 10 -15 min. after adding initial acid, prior to adding heat. Use a glass rod to break the sample surface to allow evaporation and prevent superheating below the oil surface which can cause the sample to boil and blow out of the tube.
liquors	0.5 g	Includes black, green, white, and pink liquors. For black liquors, use 0.5 g equivalent tappi solids. Note that black liquors require this method when analyzing for: Ag, As, Bi, Co, Cu, Mo, Ni, Pb, Sb, Se, Sn, and Ti.
pulps	0.5 g	For “wet” samples, use 0.5 g equivalent solids (up to 5 g of sample) to maintain the QLs. Use low level HNO_3 and prep in the ICPMS lab.
paint chips	0.05 – 0.1 g	Weigh to the nearest 0.0001 gram.

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lime, lime mud, and saltcake	0.1 g	Lime = CaO Lime Mud = $CaCO_3$ saltcake = Na_2SO_4

8.3 Add 2.5 mL of DDI-water and 2.5 mL conc HNO_3, mix the slurry, and cover with a watchglass. Heat the samples in a hotblock set at 95 °C and for 10 - 15 min without boiling. Remove samples from the hotblock and allow them to cool.

8.4 Add 2.5 mL of conc HNO_3, and reflux for 30 min. Remove samples from the hotblock and allow them to cool.

8.5 Add another 2.5 mL of conc HNO_3, and reflux for 30 min to ensure complete oxidation.

8.6 Remove watchglass and evaporate to 3 - 5 mL. Do not allow the samples to go dry. Remove samples from the hotblock and allow them to cool.

NOTE: If a sample goes dry, low recoveries will result. Should this occur, discard the sample and start over with a new aliquot.

8.7 Add 1 mL of DDI-water and 1.5 mL of 30 % H_2O_2 (**caution: for samples high in organics, add less 30 % H_2O_2 and do more additions to add the 4.5 mL total**). Cover with a watchglass and heat in the hotblock to start the peroxide reaction. Ensure that losses do not occur from the effervescence. Heat until effervescence is minimal. Remove samples from the hotblock and allow them to cool.

8.8 Add another 1.5 mL 30 % H_2O_2 and heat in the hotblock until the effervescence is minimal. Remove samples from the hotblock and allow them to cool.

8.9 Add a final 1.5 mL 30 % H_2O_2 and heat in the hotblock until the effervescence has subsided to complete the peroxide reaction. Remove samples from the hotblock and allow them to cool, unless prepping pulp samples for ICPMS. For pulp samples for ICPMS, remove watchglass and evaporate to 1 - 2 mL.

8.10 Add 2.5 mL of conc HCl and 5 mL of DDI-water and reflux for 15 min, unless prepping pulp samples for ICPMS. For pulp samples for ICPMS, add 0.5 mL of 1:1 HCl and 5 mL of DDI-water, cover with watchglass, and reflux for 15 min. Remove samples from the hotblock and allow them to cool.

8.11 Dilute to a 50-mL final volume with DDI-water, cap, and mix by inverting the tubes 4 - 5 times. Allow the particulates to settle out before analysis, generally overnight. The digestate has an approximate acid concentration of 5 % HNO_3 and 5 % HCl, unless pulp samples for ICPMS which have an approximate acid concentration of 1 % HNO_3 and 0.5 % HCl. The sample is now ready for analysis.

9.0 QUALITY CONTROL

9.1 A reagent blank must be processed with each analytical batch to detect contamination. The concentration of analyte in the blank should be less than the quantitation limit (QL) for each element.

9.2 A laboratory control sample (LC), from the list below, must be processed with each analytical batch. The results are logged in a control chart to determine accuracy and precision

laboratory control sample	comments
CRMSAB	High-Purity Standards, Sand B, lot 690703, cat# CRM-SA-B.

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for all samples except noted in this table	Weigh 0.5 g and spike per spike sheet.
Oil Reference - 2005 for oil samples	Oil reference. Weigh 0.5 g and spike per spike sheet.
GP01 for pulp samples	Grande Prairie Reference Pulp, SWD Kraft, Lot 59960 Weigh 0.5 g and spike per spike sheet.
SRM1579a for Pb in paint samples	NIST Powdered Lead-Based Paint reference. Weigh 0.05 g.

9.3 A duplicate samples should be processed with each analytical batch to indicate the reproducibility of a sample.

9.4 A spiked samples should be processed with each analytical batch to determine sample matrix effects. The spike is added after the sample is aliquoted and before any acid is added.

For RCRA metals in soils, spike per sample type, per SR. Duplicates are not required unless there is no other duplicate in the batch for the elements to be reported.

9.5 QC results should be within the respective established QC limits.

10.0 PRECISION AND ACCURACY

10.1 Precision and accuracy are sample dependent and may vary as the sample matrix varies.

10.2 Laboratory control samples (LC) are charted to set the expected precision and accuracy.

10.3 Practical quantitation limits (PQLs) are determined from the instrument detection limits (IDLs) and/or method detection limits (MDLs) pertaining to the specific instrument.

11.0 KEY WORDS

3050, AM E-3050, AM 1-3050, digestion, EPA, SW-846, FAAS, GFAAS, ICP, metals, total, total metals, solid samples, soils, solid wastes, RCRA

12.0 REFERENCES

Test Methods for Evaluating Solid Waste (SW-846), USEPA; Office of Solid Waste, Washington D.C.; Method 3050 September 1986 and Method 3050B Revised December 1996.

13.0 REVISION HISTORY

13.1 The method was updated on 08/16/04 to change the procedure for oils by GFAAS/ICPMS, to remove the H_2SO_4 prep procedure which we no longer do, to update the estimate of analytical time, and to update the PQLs.

13.2 The method was updated on 04/25/06 to update the title, remove FAAS and GFAAS (now using ICPMS), remove the instrument PQLs (varies with prep and instrument), remove reporting and calculations (part of analysis, not the prep), reword some sections, and add a note to section 8.0 (adjusting aliquots and final volumes).

13.3 The method was updated on 10/02/06 to add the prep of pulps for ICPMS and to reword some sections.

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13.4 The method was updated on 03/02/07 to include additional safety information and to reword some sections.

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W-14

Metals Analysis by ICPMS

Process Owner (TS/PM/OM/LM) **Dan Deprez**	
	Reviewed February 28, 2007 by Christine Devine Reviewed August 13, 2007 by Christine Devine Reviewed August 15, 2008 by Christine Devine
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1.0 SCOPE

1.1 This method is used for the preparation and analysis of aqueous samples using inductively coupled argon plasma mass spectrometry (ICPMS). The instrument analysis portion of this method is used for all non-aqueous samples requiring analysis by ICPMS. This method is a modified equivalent to EPA Method 200.8, revision 5.4, of EPA/600/R-94/111, May 1994.

1.2 The ICPMS used for this method is the Thermo Elemental X Series II, purchased in 2005.

1.3 Drinking water samples are digested using this method if digestion is required, which is only if the sample has turbidity of 1 NTU or greater.

1.4 Elements typically analyzed by this method are:

Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Sn, Sr, Ti, Tl, V, Zn

2.0 SUMMARY OF METHOD

A representative aqueous sample is digested with nitric acid and hydrochloric acid and analyzed by ICPMS. Non-aqueous samples are prepared using the appropriate methods for the matrix of the sample and analyzed by ICPMS.

3.0 DEFINITIONS

3.1 **Analytical Batch (batch)** - A group of samples (usually 20, no more than 25) that are prepped together.

3.2 **Calibration Blank** - The zero standard used to calibrate an instrument.

3.3 **Calibration Standards** - Standards used to calibrate an instrument in which known concentrations of analytes produce a linear response that can be used to determine the concentration of analytes in unknown samples.

3.4 **Continuing Calibration Blank (CCB)** - The zero calibration standard that is analyzed after the CCV, except the first CCV following the calibration, which is followed by an ICV and ICB. The CCB indicates instrument drift over time and verifies that the associated samples were analyzed within an acceptable tolerance.

3.5 **Continuing Calibration Verification (CCV)** - The midpoint calibration standard that is analyzed directly after calibration, after every 10 samples (maximum), and at the end of the analysis. The first CCV after the calibration verifies that the instrument has calibrated within an acceptable tolerance. The CCVs throughout the analysis indicate instrument drift over time and verify that the associated samples were analyzed within an acceptable tolerance. This is equivalent to the Instrument Performance Check (IPC) in EPA method 200.8.

3.6 **Correction Factor (CF)** - A numerical factor used to convert the instrument result to the reportable result. The correction factor is calculated using some or all of the following: initial sample aliquot, final volume, percent solids or ash, dilutions, and elemental equations.

3.7 **Duplicate (DUP)** - A second aliquot of a sample taken during the sample prep. The DUP indicates the reproducibility of a sample with a given prep method. This is equivalent to the Laboratory Duplicate (LD) in EPA method 200.8.

3.8 **Initial Calibration Blank (ICB)** - The zero calibration standard that is analyzed after the calibration (after the ICV). The ICB verifies that the instrument calibrated within an acceptable tolerance.

3.9 **Initial Calibration Verification (ICV)** - A standard made from a different source and at a different concentration than the calibration standards that is analyzed after the calibration (after the CCV). The ICV verifies that the calibration standards are within an acceptable tolerance. This is equivalent to the Quality Control Sample (QCS) in EPA method 200.8.

3.10 **Internal Standard (IS)** - A standard that is added to all standards and samples during the analysis to measure and monitor analyte signal response over time. The analytes that make up the internal standard must not be a component of the standards and samples to which the internal standard is added.

3.11 **Laboratory Control Sample (LCS)** - A sample that contains a known concentration of analytes that is carried through the entire prep and analysis process. The LCS is used to determine the effectiveness of the method to detect the analytes of interest and to determine if the prep was performed within an acceptable tolerance. This is equivalent to the Laboratory Fortified Blank (LFB) in EPA method 200.8.

3.12 **Matrix Spike (MS)** - A second aliquot of a sample taken during the sample prep that is spiked with a known concentration of analytes. The MS is used to determine sample matrix effects on the analytes of interest within a given prep method. This is equivalent to the Laboratory Fortified Sample Matrix (LFM) in EPA method 200.8.

3.13 **Method Detection Limit (MDL)** - The minimum concentration of an analyte that can be measured with 99 % confidence that the concentration is greater than zero. The MDL of each analyte for a given procedure is determined from a MDL study.

3.14 **Practical Quantitation Limit (PQL)** - A practical detection limit based upon MDLs, impurities in reagents and stock standards, instrument sensitivity, and levels of contamination in the lab environment.

3.15 **Prep Blank (PB)** - A sample blank that is carried through the entire prep and analysis process to determine levels of contamination from the prep procedure. This is equivalent to the Laboratory Reagent Blank (LRB) in EPA method 200.8.

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3.16 **Quantitation Limit (QL)** - The final reporting limit, which takes into account such things as, initial aliquots, final volumes, dilutions, percent solids, and PQLs.

3.17 **Quantitation Limit Standard (QLSTD)** - A standard that contains analytes at concentrations of the instrument PQL. The QLSTD verifies that the instrument can detect analytes at the PQL concentrations. The QLSTD is analyzed at the beginning of the analysis after the Initial Calibration Blank (ICB).

4.0 INTERFERENCES

4.1 Spectral Interferences - Interferences in which the mass of the analyte of interest is overlapped by another mass.

4.1.1 Polyatomic Interferences - A spectral interference caused by the combination of two or more atomic ions whose mass is equal to an analyte of interest. For example, $^{40}Ar^{35}Cl$ interferes with ^{75}As. Polyatomic interferences are compensated for by isotope selection and by using Collision Cell Technology (CCT). Collision Cell Technology (CCT) involves the introduction of a specific gas into the hexapole lens system which will interact and breakdown the polyatomic ions in the sample being analyzed.

Current CCT gas: 7% H - 93% He mix
Current CCT isotopes: ^{51}V, ^{52}Cr, ^{53}ClO, ^{54}Fe, ^{55}Mn, ^{73}Ge, ^{75}As, ^{78}Se, ^{120}Sn (plus ^{103}Rh H2)
Current CCT internal standard: ^{103}Rh (designated in the program as ^{103}Rh H2)

4.1.2 Oxide, Hydroxide, and Hydride Interferences - A spectral interference caused by the combination of elements in the sample with ^{16}O, ^{16}OH, and H, to produce a combined mass equal to an analyte of interest. For example, $^{48}Ca^{16}OH$ interferes with ^{65}Cu. Oxide, Hydroxide, and Hydride interferences are compensated for by optimizing the instrument. A performance report is done daily to demonstrate the instrument is operating within specified parameters.

Current specifications: $^{156}CeO/^{140}Ce < 0.025$ (ratio limit)

4.1.3 Doubly Charged Ion Interferences - A spectral interference caused by a doubly positively (+) charged ion that produces a peak at half its mass which is equal to an analyte of interest. For example, $^{140}Ce^{2+}$ interferes with ^{70}Ge and ^{70}Zn. Doubly Charged Ion interferences are compensated for by optimizing the instrument. A performance report is done daily to demonstrate the instrument is operating within specified parameters.

Current specifications: $^{137}Ba^{++}/^{137}Ba < 0.04$ (ratio limit)

4.1.4 Isobaric Interferences - A spectral interference caused by different isotopes of elements in the sample whose mass is equal to an analyte of interest. For example, Ti, V, and Cr have natural isotopes at mass 50 and therefore can interfere with each other if analyzing for one of these elements at mass 50. Isobaric interferences are compensated for by selecting isotopes that do not have an overlap and by using correction equations to account for known overlaps.

Current correction equations:
$^{43}Ca = -0.00034 * {}^{88}Sr$
$^{51}V = -2.6 * {}^{53}ClO$
$^{54}Fe = -0.028 * {}^{52}Cr$
$^{60}Ni = -0.0004 * {}^{43}Ca$
$^{82}Se = -0.89 * {}^{83}Kr$
$^{111}Cd = -0.006 * {}^{95}Mo$
$^{123}Sb = -0.124 * {}^{125}Te$

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$$^{208}Pb = 1.0 * {}^{206}Pb + 1.0 * {}^{207}Pb$$

4.2 Physical Interferences - Interferences that affect the transport of the sample through the ICPMS due to the sample matrix. Physical interferences are compensated for by keeping the dissolved solids in the samples low and by using an internal standard (IS).

Current internal standard: 20 ug/L ^{45}Sc
10 ug/L ^{103}Rh (note: ^{103}Rh is used for CCT elements as ^{103}Rh H2)
10 ug/L ^{159}Tb

4.3 Memory Interferences - Interferences from samples with high concentrations of analytes whose signal carries over into the next sample being analyzed. Memory interferences are compensated for by using a monitored rinse time in which the signal of a specified mass (or masses) is measured and a sample will not be analyzed until the signal is below a set value. Also, three replicates are performed on each sample to observe if a carry over signal is affecting a sample result.

Current monitored mass signals: 23 = 100000 cps
43 = 300 cps
123 = 5000 cps

5.0 ESTIMATE OF ANALYTICAL TIME

The sample prep method should take 8 - 12 hours. ICPMS analysis can take 4 - 12 hours.

6.0 AMOUNT OF SAMPLE REQUIRED

A minimum of 50 mL of sample is required which may not allow for QC. A more desirable amount would be 500 - 1000 mL to allow for other analyses.

7.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Samples should be preserved to a pH of < 2 with HNO_3 or refrigerated at a temperature of 4 °C. Unknown samples should be considered hazardous. Vinyl gloves should be worn when handling samples. If a sample produces a noxious odor, try to do the work with the sample in a hood. Prepared samples (digestates) will be stored for a minimum of 1 month and then dumped into the disposal drum. When the disposal drum is full it will be transferred to the chemical management unit for proper disposal.

8.0 REAGENTS AND EQUIPMENT

Reagents must not contain detectable analytes of interest.

8.1 Nitric acid (concentrated), HNO_3. CAS # 7697-37-2

DANGER: Handle with care. Wear safety glasses, vinyl gloves, lab coat, and use in a hood. Conc. nitric acid is a strong, corrosive acid and liquid and vapor can cause severe burns. Carefully add acid to water with frequent or continuous stirring. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.
Harmful if inhaled and may cause delayed lung injury. If inhaled, can cause severe irritation or burns of mucous membranes and respiratory system, resulting in coughing, difficult breathing, chest pains, pneumonia, pulmonary edema, lung inflammation, unconsciousness, and may be fatal. Can cause severe ulceration.

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Skin contact can cause severe irritation, redness, pain, and severe skin burns. Concentrated acid causes deep ulcers and stain skin a yellow or yellow-brown color.
Eye contact can cause severe irritation or burns and result in permanent damage.
Long-term exposure to concentrated vapors may cause erosion of teeth.
Nitric acid is a strong oxidizer. Contact with combustible materials, flammable materials -such as wood and solvents - or powdered metals can cause fire or explosion. Reacts with most metals to produce hydrogen gas, which can form an explosive mixture with air. Keep away from strong bases, carbides, carbonates, charcoal, (hydrogen) sulfides, cyanides, combustible materials, combustible organics or organic materials, turpentine, strong reducing agents, most common metals, carbides, ammonium hydroxide, water, and alcohols.
Certain mixtures with benzene, 1,2-dichloroethane, or dichloromethane may be detonatable.
Reaction products include highly toxic and dangerous fumes of reddish oxides of nitrogen having the equivalent affects as those noted above.
Short-term exposure limit (STEL) is 10 mg/m^3 (4 ppm) and permissible exposure limit (PEL) is 5 mg/m^3 (2 ppm).

8.2 Hydrochloric acid (concentrated), HCl. CAS # 7647-01-0

WARNING: Handle with care. Wear safety glasses, vinyl gloves, lab coat, and use in a hood. Conc. hydrochloric acid is a strong, corrosive acid and liquid and vapor can cause burns. Carefully add acid to water with frequent or continuous stirring. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.
Avoid breathing vapors. Exposure can cause spasm, destruction to tissue of the mucous membranes, inflammation and edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Symptoms of exposure may include burning sensation, coughing, choking, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. In severe cases, inhalation may result in circulatory failure, and death.
Avoid skin contact. Hydrochloric acid can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and discolor skin.
Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Long-term exposure to concentrated vapors may cause erosion of teeth.
Highly reactive with strong bases, metals, metal oxides and alkali metals, hydroxides, amines, carbonates and other alkaline materials. Avoid contact with materials such as cyanides, sulfides, sulfites, and formaldehyde.
Mixing with strong acids may produce toxic products. Conc. sulfuric acid will dehydrate hydrochloric acid, releasing hydrogen chloride gas. Certain mixtures with nitric acid will give *aqua regia*, which is unstable and extremely corrosive, and gives off toxic gas as it decomposes.
OSHA PEL is 7 mg/m^3 (5 ppm)

8.3 Solution preparation

8.3.1 Solutions will be prepared following established recipes located in a solution prep log book. All solution prep will be recorded in a log book, noting any variations to a recipe as needed. If a recipe does not exist, the prep log must state the volume, lot number, and expiration date of the stock solutions used, as well as the final volume and expiration date of the prepared solution.

8.3.2 Calibration standards (includes the CCV, ICB, and CCB), QLSTD and ICV < 1 ug/mL expire after 2 weeks or when a stock solution expires, whichever comes first.

8.3.3 Calibration standards (includes the CCV, ICB, CCB) and ICV ≥ 1 ug/mL expire after 3 months or when a stock solution expires, whichever comes first.

8.3.4 Intermediate solutions ≥ 1 ug/mL created directly from stock solutions expire after 1 year or when a stock solution expires, whichever comes first.

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8.3.5 Internal standards and the daily tuning solutions have no expiration date because the accuracy of their concentration does not affect the sample result or the quality of the data.

8.3.6 The following calibration and quality control solutions are currently used for ICPMS analysis:

ICPMS calibration and quality control standards in ug/L												
elem	cal blank ICB CCB	STD2	STD3	STD4 CCV	STD5	STD6	STD7	STD8 CCV	STD9	ICV ppb	ICV ppm	QLSTD
Ag	0	1	10	50	100					40		0.5
Al	0	5	10	50	100					40		5
As	0	1	10	50	100					40		0.5
B	0	5	10	50	100					40		5
Ba	0	1	10	50	100					40		0.5
Be	0	1	10	50	100					40		0.5
Bi	0	1	10	50	100					40		1
Ca	0					1000	10000	25000	50000		40000	50
Cd	0	1	10	50	100					40		0.5
Co	0	1	10	50	100					40		0.5
Cr	0	1	10	50	100					40		0.5
Cu	0	1	10	50	100					40		0.5
Fe	0	5	10	50	100					40		5
K	0					1000	10000	25000	50000		40000	50
Li	0	1	10	50	100					40		0.5
Mg	0					1000	10000	25000	50000		40000	50
Mn	0	1	10	50	100					40		0.5
Mo	0	1	10	50	100					40		0.5
Na	0					1000	10000	25000	50000		40000	50
Ni	0	1	10	50	100					40		0.5
P	0					1000	10000	25000	50000		40000	50
Pb	0	1	10	50	100					40		0.5
Sb	0	1	10	50	100					40		0.5
Se	0	1	10	50	100					40		0.5
Sn	0	1	10	50	100					40		0.5
Sr	0	1	10	50	100					40		0.5
Ti	0	5	10	50	100					40		5
Tl	0	1	10	50	100					40		0.5
V	0	1	10	50	100					40		0.5
Zn	0	5	10	50	100					40		5

Note that the solution concentrations may be changed to improve the ICPMS analysis process.

8.4 Expired solutions will be disposed in the disposal drum. The drum is turned over to the chemical management unit for disposal when it is full.

8.5 ICPMS used for this method is the Thermo Elemental X Series II, purchased in 2005.

9.0 AQUEOUS SAMPLE PREP PROCEDURE

SAFETY: Wear safety glasses, vinyl gloves, lab coat, and work in a hood. All heating of samples will be done in a hood.

NOTE: Sample volumes will be measured using graduated labware such that the bottom of the meniscus is at the mark for the desired amount.

NOTE: Acids and reagents will be added using a dispenser set to the desired amount.

NOTE: Sample aliquots and final volumes may vary based upon the quantity of sample provided, type of sample, and client needs. The amount of acids and reagents added will be adjusted accordingly to provide the same acid concentration in the final volume.

9.1 Label hotblock tubes with the sample I.D. using a red marker or with a "G" if using preprinted labels.

9.2 Transfer a 50-mL representative aliquot of the well-mixed sample to the appropriate labeled hotblock tube using the mark on the tube to measure the aliquot.

9.3 Add 0.5 mL of conc HNO_3 and 0.5 mL of 1:1 HCl.

9.4 Place in a hotblock set at 85 - 95 °C and evaporate to 10 mL making certain that the sample does not actively boil. Remove samples from the hotblock and allow them to cool.

9.5 Cover the samples with a watchglass and reflux for 30 minutes. Remove samples from the hotblock and allow them to cool.

9.6 Dilute to a 50-mL final volume with DDI water, cap, and mix by inverting the tubes 4 - 5 times. Allow the particulates to settle out before analysis, generally overnight. The digestate has an approximate acid concentration of 1 % HNO_3 and 0.5 % HCl. The sample is now ready for analysis.

10.0 SOLID SAMPLE PREP PROCEDURE

SAFETY: Wear safety glasses, vinyl gloves, lab coat, and work in a hood. All heating of samples will be done in a hood.

NOTE: Sample volumes will be measured using graduated labware such that the bottom of the meniscus is at the mark for the desired amount.

NOTE: Acids and reagents will be added using a dispenser set to the desired amount.

NOTE: Sample aliquot and final volumes may vary based upon quantity of sample provided, type of sample, and client needs. The amount of acids and reagents added will be adjusted to provide the same acid concentration in the final volume.

10.1 Label hotblock tubes with the sample I.D. using a blue marker or with an "I" if using preprinted labels.

10.2 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh, to the nearest 0.001 g, the following amount of sample into the appropriate labeled hotblock tube:

sample type	aliquot in grams	additional instructions
soil and solid samples	0.5 g	For “wet” samples, use 0.5 g equivalent solids (up to 5 g of sample) to maintain the QLs. For RCRA, use as-received samples.

oils	0.5 g	Wait 10 -15 min. after adding initial acid, prior to adding heat. Use a glass rod to break the sample surface to allow evaporation and prevent superheating below the oil surface which can cause the sample to boil and blow out of the tube.
liquors	0.5 g	Includes black, green, white, and pink liquors. For black liquors, use 0.5 g equivalent tappi solids. Note that black liquors require this method when analyzing for: Ag, As, Bi, Co, Cu, Mo, Ni, Pb, Sb, Se, Sn, and Ti.
pulps	0.5 g	For "wet" samples, use 0.5 g equivalent solids (up to 5 g of sample) to maintain the QLs. Use low level HNO_3 and prep in the ICPMS lab.
paint chips	0.05 – 0.1 g	Weigh to the nearest 0.0001 gram.
lime, lime mud, and saltcake	0.1 g	Lime = CaO Lime Mud = $CaCO_3$ saltcake = Na_2SO_4

10.3 Add 2.5 mL of DDI-water and 2.5 mL conc HNO_3, mix the slurry, and cover with a watchglass. Heat the samples in a hotblock set at 95 °C and for 10 - 15 min without boiling. Remove samples from the hotblock and allow them to cool.

10.4 Add 2.5 mL of conc HNO_3, and reflux for 30 min. Remove samples from the hotblock and allow them to cool.

10.5 Add another 2.5 mL of conc HNO_3, and reflux for 30 min to ensure complete oxidation.

10.6 Remove watchglass and evaporate to 3 - 5 mL. Do not allow the samples to go dry. Remove samples from the hotblock and allow them to cool.

NOTE: If a sample goes dry, low recoveries will result. Should this occur, discard the sample and start over with a new aliquot.

10.7 Add 1 mL of DDI-water and 1.5 mL of 30 % H_2O_2 (**caution: for samples high in organics, add less 30 % H_2O_2 and do more additions to add the 4.5 mL total**). Cover with a watchglass and heat in the hotblock to start the peroxide reaction. Ensure that losses do not occur from the effervescence. Heat until effervescence is minimal. Remove samples from the hotblock and allow them to cool.

10.8 Add another 1.5 mL 30 % H_2O_2 and heat in the hotblock until the effervescence is minimal. Remove samples from the hotblock and allow them to cool.

10.9 Add a final 1.5 mL 30 % H_2O_2 and heat in the hotblock until the effervescence has subsided to complete the peroxide reaction. Remove samples from the hotblock and allow them to cool, unless prepping pulp samples for ICPMS. For pulp samples for ICPMS, remove watchglass and evaporate to 1 - 2 mL.

10.10 Add 2.5 mL of conc HCl and 5 mL of DDI-water and reflux for 15 min, unless prepping pulp samples for ICPMS. For pulp samples for ICPMS, add 0.5 mL of 1:1 HCl and 5 mL of DDI-water, cover with watchglass, and reflux for 15 min. Remove samples from the hotblock and allow them to cool.

10.11 Dilute to a 50-mL final volume with DDI-water, cap, and mix by inverting the tubes 4 - 5 times. Allow the particulates to settle out before analysis, generally overnight. The digestate has an approximate acid concentration of 5 % HNO_3 and 5 % HCl, unless pulp samples for ICPMS

which have an approximate acid concentration of 1 % HNO_3 and 0.5 % HCl. The sample is now ready for analysis.

11.0 ANALYSIS PROCEDURE

The instrument manual, Thermo Electron Corporation, X Series ICP-MS Getting Started Guide, is followed to run the instrument.

11.1 Check the following to ensure sufficiency for the analysis:

argon (regulator set to 95 psi at tank and 80 psi at instrument), helium-hydrogen mix (regulator set to 15 psi = 1 bar), oil in pump, 1 % HNO_3 rinse solution, space in the used rinse solution container, standards and expiration dates, internal standard, tubing condition, and paper in printer

Resolve any deficiencies.

11.2 Turn on the computer and enter the ICPMS program.

11.3 Turn on the water recirculator. Note that the recirculator is set to maintain 20 °C.

11.4 Ignite the instrument and allow at least 30 minutes for the instrument to warm up. Record the instrument settings in the daily operation logbook, ensuring that the parameters are similar to past performance. Ensure that the water recirculator is approximately 20 °C and the Peltier Power Pack Spray Chamber Cooling Unit is approximately 3 °C.

11.5 Run a performance report to verify the instrument passes the specified operating requirements. Note that a performance report is always needed in the XtY Standard Mode as it verifies the mass calibration. However, the XtY CCT KED mode only needs to be done when CCT elements are being analyzed. If the performance report fails, the problem must be resolved and a passing performance report obtained prior to continuing on with the analysis.

11.5.1 Open the performance report wizard.

11.5.2 Select the mode (XtY Standard Mode or XtY CCT KED).

11.5.3 Put the sample probe in solution Tune A (10 ug/L Ba, Be, Bi, Ce, Co, In, Li, Ni, Pb, U) and the IS (Internal Standard) probe in DDI-water.

11.5.4 Analyze the solution. Note that the analysis consists of 5 replicates of the Tune A solution.

11.6 Set up the software to analyze the desired samples and analytes, taking 3 replicates. Perform the analysis and report the results that meet the required quality control, which includes results with acceptable non-conformances.

11.7 An example of a basic ICPMS analysis of 10 samples with quality control (QC) would be as follows:

Calibration Blank
Standard 2
Standard 3
Standard 4
Standard 5
Standard 6 (for Ca, K, Mg, Na, P - high levels)

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Standard 7 (for Ca, K, Mg, Na, P - high levels)
Standard 8 (for Ca, K, Mg, Na, P - high levels)
Standard 9 (for Ca, K, Mg, Na, P - high levels)
CCV (continuing calibration verification)
CCV (continuing calibration verification) (for Ca, K, Mg, Na, P - high levels)
ICV (initial calibration verification)
ICV (initial calibration verification) (for Ca, K, Mg, Na, P - high levels)
ICB (initial calibration blank)
QLSTD (quantitation limit standard)
PB (prep blank)
LCS (laboratory control sample)
sample1
sample1DUP (duplicate)
sample1MS (matrix spike)
sample2
sample3
sample4
sample5
sample6
CCV (continuing calibration verification)
CCV (continuing calibration verification) (for Ca, K, Mg, Na, P - high levels)
CCB (continuing calibration blank)
sample7
sample8
sample9
sample10
CCV (continuing calibration verification)
CCV (continuing calibration verification) (for Ca, K, Mg, Na, P - high levels)
CCB (continuing calibration blank)

12.0 QUALITY CONTROL (QC)

The following section contains the expected quality control parameters to be met in order to report a result. Note that some results can be reported with failed QC provided that the failed criteria will not have an effect on the sample result and a satisfactory explanation is noted on the data or non-conformance memo. For example, if a CCV fails high for an element (indicating instrument drift that will bias the result to be higher) and the sample is non-detect, there is no effect to the element to be reported. When a QC parameter fails, the reason for failure should be determined so that an appropriate corrective action can be taken. Only the samples affected by the failed QC parameter need to be resolved. Reasons for failed QC may include contamination, poor lab technique, improper method, sample matrix effect, need new standards, poor calibration, instrument drift, or need instrument maintenance. The appropriate corrective action may include reprep samples (redigest), recalibrate instrument, reanalyze samples, reprep standards, dilute the samples (to remove an interference or to get the sample within the calibration range), use of a different method, instrument maintenance, a note on the data, or a non-conformance memo.

12.1 A prep blank must be processed with each analytical batch to detect contamination. The concentration of analyte in the blank should be less than the quantitation limit (QL) for each element.

12.2 A laboratory control sample (LCS) must be processed with each analytical batch. Currently the LCSW (Laboratory Control Sample Water = a spiked blank) is used for aqueous samples and

the LCS for a solid sample will vary with the method and type of sample as stated in the prep method being used. The results for each reported element in the LCSW must be 85 - 115 % of the true value and the solid reference must be within the established control limits.

12.3 A duplicate (DUP) sample should be processed with each analytical batch, at a frequency of 5 %. The duplicate indicates the reproducibility of a sample within a batch. For NPDES samples, a 10 % frequency of duplicates is required within a batch. The relative percent difference (RPD) between the sample and the duplicate should be ≤ 20 % for samples > 5 × QL. There are no limits for samples ≤ 5 × QL. Duplicates that fail to meet the criteria are assumed to be non-homogenous samples, unless there are any other indicators of cause within the analysis. A failed duplicate should be reported.

12.4 A matrix spike (MS) sample should be processed with each analytical batch, at a frequency of 5 %. The matrix spike is used to determine sample matrix effects of a sample within a batch. For NPDES samples, a 10 % frequency of spikes is required within a batch. The spike is to be added after the sample is aliquoted and before any acid is added. The matrix spike recoveries should be 70 - 130 % for samples with a spike to background ratio ≥ 5, 55 - 145 % for samples with a spike to background ratio = 1, 28 - 170 % for samples with a spike to background ratio = 0.5, and no limits for samples with a spike to background ratio ≤ 0.3. Matrix spikes that fail to meet the criteria are assumed to be caused by a non-homogenous sample or an uncorrected matrix effect, unless there are other indicators of cause within the analysis. A failed matrix spike recovery should be noted on the report.

12.5 Performance report requirements:

12.5.1 Mass Calibration verification of ^{7}Li, ^{115}In, ^{238}U:

The peak widths measured at 5 % of the peak maximum must be 0.65 - 0.85, with the optimal peak width being 0.75.

12.5.2 Sensitivity and stability results:

^{7}Li = countrate > 30,000 and %RSD < 5
^{115}In = countrate > 200,000 and %RSD < 5
^{238}U = countrate > 400,000 and %RSD < 5

^{220}Bkg (background) = countrate < 1

$^{137}Ba^{++}/^{137}Ba$ < 0.04 (ratio limit)

$^{156}CeO/^{140}Ce$ < 0.025 (ratio limit)

12.5.3 CCT requirements:

^{78}Se = countrate < 50
^{115}In = countrate > 50,000 and %RSD < 5
$^{156}CeO/^{140}Ce$ < 0.025 (ratio limit)

12.6 The internal standard (IS) response must be 60 - 125 % of the original response in the calibration blank. If a sample contains ^{45}Sc, ^{103}Rh, or ^{159}Tb, the element(s) will not be used as an internal standard. If the IS response is outside 60 - 125 % range, dilute the sample 5× and reanalyze.

12.7 The correlation coefficient (r) of the calibration must be ≥ 0.995.

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12.8 The sample results must be within the calibration range (i.e., ≥ - PQL to ≤ the highest standard).

12.9 The relative standard deviation (RSD) of the replicates of each sample must be ≤ 20 % for samples ≥ 2 × PQL. There are no limits for samples < 2 × PQL.

12.10 The continuing calibration verification (CCV) must be 90 - 110 % of the true value.

12.11 The initial calibration verification (ICV) must be 90 - 110 % of the true value. Note that a second analysis of the ICV can be done immediately if the ICV fails.

12.12 The initial calibration blank (ICB) and continuing calibration blank (CCB) must be within ± the practical quantitation limit (PQL).

12.13 The quantitation limit standard (QLSTD) must be in the established control limits.

12.14 The method detection limits (MDLs) will be determined from a MDL study. The MDLs will be used to determine the practical quantitation limits (PQLs) and reporting quantitation limits (QLs) for each element.

13.0 CALCULATIONS

13.1 The ICPMS is currently set to output results in ug/L. To convert the instrument result in ug/L to the reporting result for a sample, a correction factor (CF) is used. The correction factor is determined by the volume of sample aliquoted, the final volume, and % solids or ash. Note that if dilutions were made at the instrument, the instrument result must be multiplied by the dilution factor prior to conversion to the reporting result.

13.2 To report sample results in ug/L:

instrument result (ug/L) × prep final volume (mL) ÷ prep initial volume (mL)

13.3 To report sample results in mg/L:

sample result (ug/L) ÷ 1000

13.4 To report sample results in mg/kg, as-received basis:

instrument result (ug/L) × prep final volume (L) ÷ prep initial volume (g)

note: mg/kg = ug/g

13.5 To report sample results in mg/kg, solids or ash basis:

sample result (mg/kg) ÷ percent solids or percent ash (decimal)
NOTE: Solids basis can be Air Dried, Oven Dried (O.D.), or TAPPI Solids. For oven dried, please see test method, AM S-2540G. For TAPPI solids, please seen test method, AM T-650.

13.6 To report on a percent basis (% basis):

sample result (mg/L or mg/kg) ÷ 10,000

13.7 To calculate the relative percent difference (rpd) for the sample and duplicate:

absolute value (sample result - duplicate result) ÷ ((sample result + duplicate result) ÷ 2) × 100

13.8 To calculate the % matrix spike (MS) recovery:

((spike result - sample result) ÷ spike added) × 100

13.9 To calculate the spike to background ratio for the matrix spike (MS):

spike added ÷ sample result

14.0 REPORTING

14.1 Samples will be reported down to a quantitation limit (QL). The QL is determined by multiplying the practical quantitation limit (PQL) by the same correction factor (CF) that was used to report the sample. Note that the reported result will not contain more significant figures than the QL and will contain no more than 3 significant figures.

14.2 Current Practical Quantitation Limits (PQLs):

^{107}Ag, ^{109}Ag, ^{75}As, ^{137}Ba, ^{9}Be, ^{111}Cd, ^{59}Co, ^{52}Cr, ^{65}Cu, ^{7}Li, ^{55}Mn, ^{95}Mo, ^{60}Ni, ^{208}Pb, ^{123}Sb, ^{78}Se, ^{82}Se, ^{118}Sn, ^{88}Sr, ^{205}Tl, ^{51}V = 0.5 ug/L

^{209}Bi = 1 ug/L

^{27}Al, ^{10}B, ^{54}Fe, ^{49}Ti, ^{66}Zn = 5 ug/L

^{43}Ca, ^{39}K, ^{25}Mg, ^{23}Na, ^{31}P = 50 ug/L

15.0 KEY WORDS

200.8, AM E-200.8M, ICPMS, EPA, metals, water

16.0 REFERENCES

16.1 Methods for the Determination of Metals in Environmental Samples, Supplement I (EPA/600/R-94/111), USEPA; Office of Research and Development, Washington D.C. 20460; May 1994.

16.2 Thermo Electron Corporation X Series ICP-MS Getting Started Guide, Issue 1.3: January 2004, Reference Number: S419MA.

17.0 REVISION HISTORY

17.1 The method was updated on 07/10/07 to add borders to the standards table, to state that the CCV is equivalent to the IPC, and to state that the QLSTD is analyzed after the ICB.

17.2 Methods specified in 13.5; equipment added to 8.5; typos corrected.

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W-15

Capillary GC/MS for Semivolatile Organics

Process Owner (TS/PM/OM/LM) **Randy Eatherton**	
Assignment	Reviewed July 18, 2003 by Christine Devine Reviewed October 26, 2005 by Christine Devine Reviewed July 20, 2007 by Christine Devine
Electronic only unless colored "Controlled" stamp is present	

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1.0 SCOPE

1.1 This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from solid waste matrices, soils, sediments and ground water. It is based on the EPA's SWA-846 method 8270C and the Puget Sound Estuary Protocol (marine sediments). Direct injection of a sample may be used in limited applications. The method is normally used to determine the compounds in Table 1. Additional compounds that are less frequently determined are listed in Table 2.

1.2 The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent concentration. Also, chromatography is poor. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution and photochemical decomposition. N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenyl amine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.3 The practical quantitation limit (PQL) of this method for determining an individual compound is approximately 330 µg/kg (wet weight) for soil/sediment samples, and 10 µg/L for ground water samples (see Table 5 for PQLs). PQLs will be higher depending upon dilution required and the moisture content of the sample.

1.4 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

Prior to using this method, the samples should be prepared for chromatography using the appropriate sample preparation and cleanup methods. This method describes chromatographic conditions that will allow for the separation of the compounds in the extract.

2.1 Water samples will be prepared utilizing continuous liquid/liquid extraction AC G-3520.

2.2 Soil samples will be prepared utilizing ultrasonic extraction AC G-3550.

2.3 Sediment samples will be prepared utilizing ultrasonic extraction AC G-3550 or soxhlet extraction AC G-3540. Sediment samples may require larger sample sizes and a modified calibration curve to achieve the required detection limits. Marine Sediment "PSEP" sample preparation method modifications are detailed in Appendix 3.

2.4 All soil and sediment samples will be cleaned up by gel permeation chromatography, AC G-3640-GPC.

2.5 Sediment samples may require a silica gel clean-up procedure, AC G-3630 if the analysis is for polyaromatic hydrocarbons only.

3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for chromatographic interferences caused by the preparative steps performed prior to analysis or instrumental procedures. Common sources of interferences include solvents, reagents and GPC carry-over from contaminated samples.

3.2 Contamination by GC carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.0 ESTIMATE OF ANALYTICAL TIME

4.1 Tuning and initial calibration require approximately 6 hours of instrument time prior to analysis of samples. If a valid calibration is available, approximately 2 hours is required for tuning and calibration verification.

4.2 Sample analysis requires approximately 1 hour of instrument time per sample. Data work up and reporting varies depending on sample type and reporting requirements. 1 to 2 hours should be anticipated per sample.

5.0 AMOUNT OF SAMPLE

5.1. Water samples require 1 liter of sample. An additional liter is required for each duplicate, matrix-spike or matrix-spike duplicate.

5.2 Soil samples require approximately 50 grams for sample extraction, pH determination and moisture determination. An additional 30 grams is required for each duplicate, matrix spike or matrix spike duplicate.

5.3 Sediment samples require the same amount of sample as soils unless low detection limits are required, such as for Puget Sound Protocol. Marine Sediment "PSEP" sample amount requirements are detailed in Appendix 3.

6.0 SAMPLE EXTRACT HANDLING AND STORAGE

6.1 Store sample extracts at 4°C until analysis.
6.2 Store sample extracts for 365 days following analysis at -10° to -20° C.

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6.3 Analyze sample extracts within 35 days.

7.0 APPARATUS AND MATERIALS

7.1 Gas chromatograph/mass spectrometer system – Hewlitt Packard 5972 MSD, capable of scanning from 35 to 500 amu every 1 sec., using 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when 1µL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

7.2 Gas chromatograph - Hewlett Packard 5890 Series II with Hewlett Packard 5973A autosampler equipped with Split/Splitless injector.

7.3 Syringe - Hamilton 701ASN, 10µL.

7.4 Column - 30 m x 0.25 mm i.d., 0.25-µm film thickness silicone-coated, fused-silica capillary column (J&W Scientific DB-5MS is preferred but equivalent columns are acceptable).

7.5 Data system - Data analysis performed using Chemserver software on a HP/UX J5600 computer. Final data reports are generated in US-EPA CLP format (OLM 3.0) using Thruput Target and Envision software.

8.0 REAGENTS

8.1 All solvents used will be pesticide-residue grade or better.

8.2 Standard solutions - Standard solutions are purchased as certified solutions.

8.2.1. Commercially certified stock standards are used with the exception of alpha-terpineol. See standards preparation AC G-ORGSTD for standard preparation procedures. If the compound purity is assayed to be 96% or greater when stock solutions are prepared, the mass may be used without correction to calculate the concentration of the stock standard.

8.2.2. Secondary standard solutions will be stored at 4°C and protected from light. Stock standard solutions will be stored at -10° to -20°C and protected from light. All standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards or samples from them. The level of the solution in the container should be marked after each use so any evaporation can be detected.

8.2.3. The stock standard solutions must be replaced according to the manufactures expiration date. Working standards are replaced after 1 year or at the stocks expiration date (whichever is less), or sooner, if comparison with quality control check-samples indicates a problem.

8.2.4. Internal standard solutions - The internal standards used are listed in Table 3. The internal standard spiking solution will contain each standard at a concentration of 2,000 ng/µL. Each sample extract undergoing analysis should be spiked with 1 µL of the internal standard solution per 100 µL of sample extract, resulting in a concentration of 20 ng/µL of each internal standard with a 1000-µL final extract volume.

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8.2.5. GC/MS tuning standard consists of a methylene chloride solution containing 25 ng/µL of decafluorotriphenylphosphine (DFTPP).

8.2.6. Calibration standards

a. A minimum of five calibration standards should be prepared.

b. The normal concentrations of the calibration standards will be 10 µg/mL, 25 µg/mL, 40 µg/mL, 60 µg/mL and 80 µg/mL. If decreased detection limits are required, the additional calibration standards may be prepared down to a minimum concentration of 1 µg/mL.

c. Each standard contains each analyte for detection by this method (e.g., all of the compounds listed in Table 1 will be included). Compounds in Table 2 and selected additional compounds may be included if method performance criteria can be met.

d. Each aliquot of calibration standard is spiked with 1 µL of the internal standard solution per 100 µL of solution prior to analysis.

8.2.7. Surrogate standards - The six surrogate compounds used are listed in Table 3. 2-Chlorophenol-d4 and 1,2-Dichlorobenzene-d4 are also listed and may be used as alternate surrogate standards. The acid surrogates (phenolic) are spiked to achieve a final concentration of 75 µg/mL. The base/neutral surrogates are spiked to achieve a final concentration of 50 µg/mL. The extraction procedures provide instruction for preparing and adding surrogate spikes.

8.2.8. Laboratory control spike compounds (LCS) and matrix-spike compounds are listed in Table 11. Full-target compound laboratory control spikes (FLCS) and matrix spike compounds are listed in Table 1 (Due to the cost involved, FLCS are not analyzed unless requested by the client). Extraction procedures provide instruction for preparing and adding spike solutions.

9.0 PROCEDURE

See Section 2, Summary of Method, for appropriate extraction methods.

9.1 GC/MS operating conditions:

9.1.1. Mass range: 35-500 amu

9.1.2. Scan time: 0.9 sec/scan; 0.1 sec interscan time

9.1.3. Initial column temperature and hold time: 40° C for 1 minute

Note:If N-nitrosodimethylamine is to be determined, the initial temperature must be reduced to 35° C and the initial hold time must be extended to approximately 5 minutes.

9.1.4. Column temperature program: 40-320° C at 8° C/min

9.1.5. Final column temperature hold: 1-3 minutes @ 320° C

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9.1.6. Injector temperature: 270° C

9.1.7. Transfer line temperature: 250° C

9.1.8. Source temperature: 180° C

9.1.9. Injector: Grob-type, splitless

9.1.10. Sample volume: 1 µL

9.1.11. Carrier gas: Helium at 30 cm/sec

9.2 Instrument Tune - GC/MS system must be hardware-tuned to meet the criteria in Table 5 for a 50-ng injection of DFTPP. Analyses may not begin for any standards or samples until all these criteria are met. Background subtraction should be straightforward and designed only to eliminate column bleed or instrument background ions. It is suggested that the spectra for DFTPP be generated using a scan range ± 1 scan number from the scan number closest the total ion current maximum for DFTPP. Mass spectrometer conditions must be identical for subsequent analysis of standards, samples, QC samples and blanks associated with the DFTPP analysis.

9.3 Injector and Column maintenance

9.3.1. The injector sleeve should be clean and compound breakdown or adsorption should be minimized.

9.3.2. The capillary column should be in good condition, with minimal tailing noted for reactive compounds, such as pentachlorophenol.

9.4 The internal standards should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the internal standards specified in Table 3 to calculate compound concentrations.

9.5 Initial Calibration

9.5.1. Tune instrument as outlined in Section 9.2 above.

9.5.2. Analyze 1 µL of five or more calibration standards (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 1). Figure 1 shows a chromatogram of a calibration standard containing base/neutral and acid analytes.

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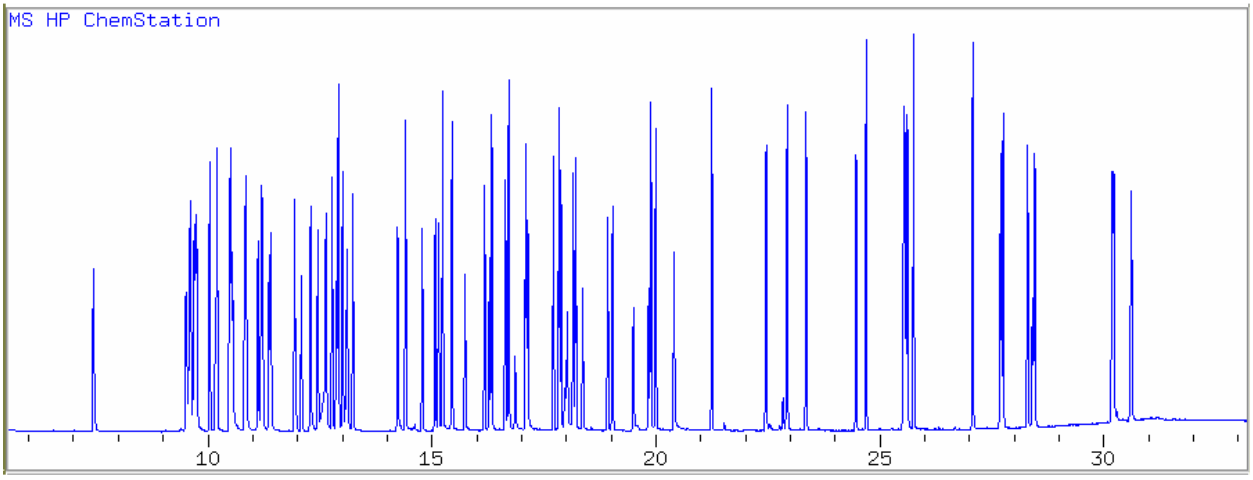

Figure 1

9.5.3. Calculate response factors (RFs) for each compound as shown below:

RF = (AxCis)/(AisCx)

where:

Ax = Area of the characteristic ion for the compound being measured.

Ais = Area of the characteristic ion for the specific internal standard.

Cx = Concentration of the compound being measured (µg/µL).

Cis = Concentration of the specific internal standard (µg/µL).

9.5.4.

a. Calculate the average RF and the percent relative standard deviation for each compound. If the RSD of any target compound is 15 % or less, the average relative response factor may be used for quantitation. If the RSD is greater than 15 %, Option 1 must be used:

Option 1 – Calculate a linear calibration using least-squares regression for each compound exceeding the 15 % RSD criteria. The correlation coefficient, r, must be greater than or equal to 0.99.

If the correlation coefficient, r, is less than 0.99, Option 2 must be used:

Option 2 – Calculate a non-linear calibration using a quadratic- or third-order polynominal regression for each compound exceding the 15 % RSD criteria. The coefficient of the determination, COD, must be greater than or equal to 0.99. **Note: SW-846 requires six calibration standards for second-order polynominals and seven calibration standards for third-order polynominals**.

If the coefficient of the determination, COD, is less than 0.99, Option 3 must be used:

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Option 3. - Calculate the mean of the RSD values for all analytes in the calibration. If it is less than or equal to 15%, the initial calibration is valid. Note: A summary report indicating compounds that exceeded the 15% RSD criteria and the results of the mean RSD calculation is required.

b. The %RSD for each individual Calibration Check Compound (CCC) (see Table 6) must be less than 30%. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units. If the CCCs are not included in the project compound list, all compounds must meet the 30% RSD criteria.

9.5.5. A system-performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compounds (SPCCS) are: N-nitroso-di-n-propylamine; hexachlorocyclo-pentadiene; 2, 4-dinitro-phenol; and 4-nitrophenol. The minimum acceptable average RF for these compounds is 0.050. These SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

9.6 Daily GC/MS calibration

9.6.1. Prior to analysis of the daily calibration check, the GC/MS tuning standard must be analyzed and meet performance criteria as in 9.2.

9.6.2. A 50-µg/mL calibration standard containing each compound of interest, including all required surrogates, must be performed every 12 hours immediately following DFTPP during analysis. Compare the response factor data from the standards every 12 hours with the average response factor from the initial calibration for a specific instrument as per the SPCC, and CCC criteria in 9.6.3 and 9.6.4.

9.6.3. System Performance Check Compounds (SPCCs): A system performance check must be made during every 12-hour shift. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. The minimum RF for semivolatile SPCCs is 0.050. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column and active sites in the column or chromatographic system. This check must be met before analysis begins.

9.6.4. Calibration Check Compounds (CCCs): After the system performance check is met, CCCs listed in Table 6 are used to check the validity of the initial calibration. For compounds using an average response factor calculate the percent difference using:

$$\%\,\text{Difference} = \frac{(RF_I - RF_C)}{\overline{RF_I}} \times 100$$

where:

$\overline{RF_I}$ = average response factor from initial calibration.

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RF_c = response factor from current verification check standard.

For compounds using a linear or non-linear calibration curve, calculate the percent drift using:

$$\%\,\text{Drift} = \frac{(\textit{Calculated concentration - Theoretical concentration})}{\textit{Theoretical concentration}} \times 100$$

Where: the calculated concentration is determined using the mean calibration factor.

If the percent difference for each CCC is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% difference for any one CCC), corrective action must be taken. If the CCCs are not included in the project compound list, all compounds must meet the 20% RSD criteria. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration must be generated. This criterion must be met before sample analysis begins.

9.6.5. The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last daily calibration check, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the Extracted Ion Current Profile (EICP) area for any of the internal standards changes by a factor of two (-50% to +100%) from the most recent initial calibration midpoint standard, the mass spectrometer must be inspected for malfunctions and appropriate corrections must be made.

9.7 GC/MS analysis

9.7.1. The extract may be screened on a GC/FID or GC/PID using the same type of capillary column. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

9.7.2. Spike the sample extract or an aliquot of the sample extract with 1 µL of the internal standard solution (8.2.4) per 100 µL of sample extract, just prior to analysis.

9.7.3. Analyze the sample extract by GC/MS using the same conditions as were used for the initial and continuing calibration.

9.7.4. If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 20 ng/µL of each internal standard in the extracted volume. The diluted extract must be re-analyzed.

9.7.5. After analysis, store the extracts at 4° C, protected from light, in vials equipped with unpierced Teflon lined septa.

9.8 Data interpretation

9.8.1. Qualitative analysis

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a. An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference should be obtained on the GC/MS using the same operating conditions and DFTPP tune conditions as are used for sample analysis. These standard reference spectra may be obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC relative retention time (RRT) as the standard component, and (2) correspondence of the sample component and the standard component mass spectrum.

b. The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same 12 hours as the sample. The RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

c. All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

d. The relative intensities of ions specified should agree within ± 20% between the standard and sample spectra.

e. If these criteria are not met, but, in the opinion of the mass spectral specialist, the analyte is detected in the sample, the analyte will be reported.

f. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral specialist assign a tentative identification. Guidelines for making tentative identification are:

i. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

ii. The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)

iii. Molecular ions present in the reference spectrum should be present in the sample spectrum.

viii. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

v. Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

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9.8.2. Quantitative analysis

a. When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standards to be used for each compound are listed in Table 7. If the compound does not appear in Table 7, the internal standard with a retention time nearest the retention time of that of a given analyte should be used.

b. Calculate the concentration of each identified analyte in the sample as follows:

Water

$$\text{Concentration}(\mu\text{g/L}) = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\overline{RF})(V_o)(V_i)}$$

where:

A_X = Area of characteristic ion for the compound being measured.

I_s = Amount of internal standard injected (ng).

V_t = Volume of total extract, taking into account dilutions (i.e.; a 1 into 10 dilution of a 1-mL extract will mean Vt = 10,000 µL. If half the base/neutral extract and half the acid extract are combined, Vt = 2,000).

A_{is} = Area of characteristic ion for the internal standard.

$\overline{RF}$ = Average Response factor for compound being measured (See IX.F.4).

V_o = Volume of water extracted (mL).

V_i = Volume of extract injected (µL).

Sediment/Soil Sludge (dry-weight basis)

$$\text{Concentration}\,(\mu\text{g/kg}) = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\overline{RF})(V_i)(W_s)(D)}$$

where:

A_X, I_s, V_t, A_{is}, $\overline{RF}$, V_i - Are the same as for water.

W_S = Mass of sample extracted or diluted, in grams.
D = (100 - % moisture)/100, or 1 for a wet-weight (as-received) basis.

c. Where applicable, an estimate of concentration for non-calibrated components in the sample (tentatively identified compounds) should be made. The formulas given above should be used with the following modifications: The areas A_x and

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A_{is} should be from the total ion chromatograms and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

d. Report results without correction for recovery data. When duplicates and spiked samples are analyzed, report all data obtained with the sample results.

e. Report results less than 10 parts-per-billion (PPB) to one significant figure. Report results greater than 10 PPB to two significant figures.

10.0 QUALITY CONTROL

10.1 Each day that an analysis is performed, DFTPP tune standard and the daily calibration standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are:

10.1.1. Does the air leak spectrum look ok? Is there evidence of an injector/septum leak?

10.1.2. Does FC43 calibration gas look normal? Are the ratios of m/z's 69, 131, 219 and 502 correct? Does the resolution look normal?

10.1.3. Does DFTPP meet criteria in Table 5 easily?

10.1.4. Do the chromatograhic peaks look normal for DFTPP and for the daily calibration standard?

10.1.5. Is the response obtained comparable to the response from previous DFTPP analyses and daily calibration analyses?

If any of the above do not appear normal, make appropriate corrections before attempting to analyze samples.

10.2 Required instrument QC before samples may be analyzed.

10.2.1. The GC/MS system must be tuned to meet the DFTPP specifications in Table 5.

10.2.2. Before processing any samples, the analyst should demonstrate, through the analysis of the method blank associated with the samples, that interferences from the analytical system and sample extraction are under control. Each set of samples that is extracted will be extracted with a method blank. The blanks must be carried through all stages of sample preparation and measurement with the samples.

10.2.3. There must be an initial calibration of the GC/MS system as specified in 9.5.

10.2.4. The GC/MS system must meet the SPCC criteria specified in 9.6.3 and the CCC criteria in 9.6.4, each 12-hour period.

10.3 To establish the ability to generate acceptable accuracy and precision, the analyst must perform an initial demonstration of precision and accuracy (IPAR) as outlined in SOP, OQ-TRAIN.

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10.3.1. A quality control (QC) reference sample concentrate is required containing each analyte at a concentration of 100 µg/mL in acetone. The QC reference sample concentrate may be prepared from pure standard materials or purchased as certified solutions. If prepared by the laboratory, the QC reference sample concentrate must be made using stock standards prepared independently from those used for calibration.

10.3.2. Using a gas tight syringe, prepare QC reference samples at a concentration of 100 µg/L by adding 1.00 mL of QC reference sample concentrate to each of four 1-L aliquots of water.

10.3.3. Analyze the well-mixed QC reference samples according to the method, beginning in section 9 with extraction of the samples.

10.3.4. Calculate the average recovery (x) in µg/L, and the standard deviation (s) of the recovery in µg/L, for each analyte of interest using the four results.

10.3.5. For each analyte, compare s and x with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 9. If s and x for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual x falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

NOTE: The large number of analytes in Table 9 present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are analyzed.

10.3.6. When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must locate and correct the source of the problem and repeat the test for all analytes of interest beginning with 9.6. Repeated failure may require recalibration, beginning with 9.5.

10.4 The laboratory must analyze a reagent blank for each analytical batch (up to a maximum of 20 samples/batch, except for samples required under regulation which need QC every 10 samples), and should analyze a Laboratory Control Spike (LCS) for each analytical batch. A matrix spike and matrix spike duplicate should be analyzed for each 20 samples, except for samples required under regulation. Regulatory samples need QC every 10 samples. Matrix spikes may not be analyzed with each extraction batch.

Compare the percent recovery (p_s) for each analyte in the LCS with the laboratory generated QC charts (generated in 10.5.1a). If the recovery of any analytes of interest falls outside the designated range, the laboratory performance for that analyte is judged to be out of control, and the problem identified and corrected. The analytical result for that analyte in the unspiked sample is suspect and, if possible, the samples associated with the LCS should be reanalyzed.

10.5 As part of the QC program for the laboratory, method accuracy and precision for each matrix studied must be assessed and records must be maintained.

10.5.1. To determine acceptable accuracy and precision limits for LCS samples the following procedure should be performed.

a. After the analysis of five LCS (of the same matrix), calculate the average percent recovery (p) and the standard deviation of the percent recovery (s_p). Plot p, with

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a percent recovery interval of p - 2 s_p to p + 2 s_p, with s the control limit and p - 3 s_p to p + 3 s_p as the action limit. (For example, if p = 90% and s_p = 10%, the control limit is 70-110% and the action limit is 60-120%.) Update the recovery charts for each analyte on a regular basis (e.g.; after each ten new measurements).

b. Only the twenty most recent LCS should be used to generate the QC limits.

c. For aqueous and soil matrices, these laboratory established LCS action limits may, if applicable, be compared with the control limits listed in Table 11.

10.5.2. To determine acceptable accuracy and precision limits for surrogate standards the following procedure will be performed.

a. After the analysis of five method blanks (of the same matrix), calculate the average percent recovery (p) and the standard deviation of the percent recovery (s_p). Plot p with a percent recovery interval of p - 2 s_p to p + 2 s_p as the control limit and p - 3 s_p to p + 3 s_p as the action limit. (For example, if p = 90% and s_p = 10%, the control limit is 70-110% and the action limit is 60-120%.) Update the recovery charts for each analyte on a regular basis (e.g.; after each twenty new measurements).

b. For aqueous matrices, these laboratory established action limits should, if applicable, be compared with the control limits listed in Table 10. The limits given are multi-laboratory performance based limits from EPA 8270, Rev 1, for soil and aqueous samples. Therefore, the single-laboratory limits established in the recovery chart must fall within those given in Table 10 for these matrices.

c. If recovery is not within limits, the following procedures are required. Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance. Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem. If possible, re-extract and re-analyze the sample if none of the above are a problem or note the recovery problem in the report narrative.

10.6 An MDL Study will be performed at least once per year as outlined in SOP, AQ G-MDL.

11.0 REFERENCES

11.1 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Method 625,' October 26, 1984.

11.2 U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, July 1985, Revision.

11.3 Provost, L.P. and R.S. Elder, "Interpretation of Percent Recovery Data," American Laboratory, 15, 58-63, 1983.

11.4 Eichelberger, J.W., L.E. Harris, and W.L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry Systems," Analytical Chemistry, 47, 995-1000, 1975.

11.5 "Method Detection Limit for Methods 624 and 625," Olynyk, P., W.L. Budde, and J.W. Eichelberger, Unpublished report, October 1980.

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11.6 "Interlaboratory Method Study for EPA Method 625-Base/Neutrals, Acids, and Pesticides," Final Report for EPA Contract 68-03-3102 (in preparation).

11.7 Burke, J.A. 'Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects,' Journal of the Association of Official Analytical Chemists, 48, 1037, 1965.

11.8 Lucas, S.V.; Kornfeld, R.A. "GC-MS Suitability Testing of RCRA Appendix VIII and Michigan List Analytes "; U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268, February 20, 1987, Contract No. 68-03-3224.

11.9 Engel, T.M.; Kornfeld, R.A.; Warner, J.S.; Andrews, K.D. 'Screening of Semivolatile Organic Compounds for Extractability and Aqueous Stability by SW-846, Method 3510"; U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268, June 5, 1987, Contract 68-03-3224.

11.10 U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, OLM1.8.

11.12 U.S. EPA Method 8270, Revision 1, December 1987.

12.0 REVISION HISTORY

12.1 7/17/07 – Updated references to SOPs and made small corrections to directions regarding which section to which one should refer.

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Table 1 -- List of characteristic ions of semi-volatile compounds

Compound	CAS#	Primary Ion	Secondary Ions
Acenaphthene	83-32-9	154	153, 152
Acenaphthylene	208-96-8	152	151, 153
Anthracene	120-12-7	178	176, 179
Benz(a)anthracene	56-55-3	228	229, 226
Benzo(b)fluoranthene	205-99-2	252	253, 125
Benzo(k)fluoranthene	207-08-9	252	253, 125
Benzo(g,h,i)perylene	191-24-2	276	138, 277
Benzo(a)pyrene	50-32-8	252	253, 125
Bis(2-chloroethoxy)methane	111-91-1	93	95, 123
Bi-s(2-chloroethyl)ether	111-44-4	93	63, 95
Bis(2-chloroisopropyl)ether	39638-32-9	45	77, 121
Bis(2-ethylhexyl)phthalate	117-81-7	149	167, 279
4-Bromophenyl phenyl ether	101-55-3	248	250, 141
Butyl benzyl phthalate	85-68-7	149	91, 206
4-Chloroaniline	106-47-8	127	129
4-Chloro-3-methylphenol	59-50-7	107	144, 142
2-Chloronaphthalene	91-58-7	162	127, 164
2-Chlorophenol	95-57-8	128	64, 130
4-Chlorophehyl phenyl ether	7005-72-3	204	206, 141
Chrysene	218-01-9	228	226, 229
Dibenz(a,h)anthracene	53-70-3	278	139, 279
Dibenzofuran	132-64-9	168	139
Di-n-butylphthalate	84-74-2	149	150, 104
1,2-Dichlorobenzene	95-50-1	146	148, 111
1,3-Dichlorobenzene	541-73-1	146	148, 111
1,4-Dichlorobenzene	106-46-7	146	148, 111
3,3-Dichlorobenzidine	91-94-1	252	254, 126
2,4-Dichlorophenol	120-83-2	162	164, 98
Diethyl phthalate	84-66-2	149	177, 150
2,4-Dimethylphenol	105-67-9	122	107, 121
Dimethyl phthalate	131-11-3	163	194, 164
4,6-Dinitro-2-methylphenol	534-52-1	198	51, 105
2,4-Dinitrophenol	51-28-5	184	63, 154
2,4-Dinitrotoluene	121-14-2	165	63, 89
2,6-Dinitrotoluene	606-20-2	165	63, 89
1,2-Diphenylhydrazine	122-66-7	77	105, 182
Di-n-octylphthalate	117-84-0	149	167, 43
Fluoranthene	206-44-0	202	101, 203
Fluorene	86-73-7	166	165, 167
Hexachlorobenzene	118-74-1	284	142, 149
Hexachlorobutadiene	87-68-3	225	223, 227
Hexachlorocyclopentadiene	77-47-4	237	235, 272
Hexachloroethane	67-72-1	117	201, 199
Indeno(1,2,3-cd)pyrene	193-39-5	276	138, 227
Isophorone	78-59-1	82	95, 138
2-Methylnaphthalene	91-57-6	142	141

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Compound	CAS#	Primary Ion	Secondary Ions
2-Methylphenol	95-48-7	108	107, 79
4-Methylphenol	106-44-5	108	107, 79
Naphthalene	91-20-3	128	129, 127
2-Nitroaniline	88-74-4	65	92, 138
3-Nitroaniline	99-09-2	138	108, 92
4-Nitroaniline	100-01-6	138	108, 92
Nitrobenzene	98-95-3	77	123, 65
2-Nitrophenol	88-75-5	139	109, 65
4-Nitrophenol	100-02-7	139	109, 65
N-Nitrosodiphenylamine	86-30-6	169	168, 167
N-Nitrosodi-n-propylamine	621-64-7	70	42, 101, 130
Pentachlorophenol	87-86-5	266	264, 268
Phenanthrene	85-01-8	178	179, 176
Phenol	108-95-2	94	65, 66
Pyrene	129-00-0	202	200, 203
1,2,4-Trichlorobenzene	120-82-1	180	182, 145
2,4,5-Trichlorophenol	95-95-4	196	198, 200
2,4,6-Trichlorophenol	88-06-2	196	198, 200

Table 2 -- List of characteristic ions of semi-volatile compounds

Compound	CAS#	Primary Ion	Secondary Ions
Benzidine	92-87-5	184	92, 185
Benzoic acid	65-85-0	122	105, 77
Benzyl alcohol	100-51-6	108	79, 77
N-Nitrosodimethylamine	62-75-9	42	74, 44
3-Methylphenol	108-39-4	108	107, 79
Pyridine	110-86-1		

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Table 3 - Internal Standards and Surrogates

Compound	Primary Ion	Secondary Ions
Internal Standards		
1,4-Dichlorobenzene-d4	152	115
Naphthalene-d8	136	68
Acenaphthene-d10	164	162, 160
Phenanthrene-d10	188	94, 80
Chrysene-d12	240	120, 236
Perylene-d12	264	260, 265
Surrogates		
Phenol-d5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
Nitrobenzene-d5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl	244	122, 212
2-Chlorophenol-d4(alternate)	132	68, 134
1,2-Dichlorobenzene-d4(alternate)	152	115, 150

Table 4 - PQLs FOR SEMIVOLATILE ORGANICS[b]
(EPA METHOD 8270, 1987 Rev. 1)

	Practical Quantitation Limits[b]	
Semivolatiles	Ground Water µg/L	Low Soil/ Sediment µg/kg
Acenapthene	10	660
Acenaphthylene	10	660
Anthracene	10	660
Benz(a)anthracene	10	660
Benzo(b)fluoranthene	10	660
Benzo(k)fluoranthene	10	660
Benzoic acid	50	3300
Benzo(g,h,i)perylene	10	660
Benzo(a)pyrene	10	660
Benzyl alcohol	20	1300
Bis(2-chloroethoxy) methane	10	660
Bis(2-chloroethyl) ether	10	660
Bis(2-chloroisopropyl) ether	10	660
Bis(2-ethylhexyl)phthalate	10	660
4-Bromophenyl phenyl ether	10	660
Butyl benzyl phthalate	10	660
4-Chloroaniline,	20	1300
4-Chloro-3-methylphenol	20	1300
2-Chloronaphthalene	10	660
2-Chlorophenol	10	660

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Semivolatiles	Ground Water µg/L	Low Soil/ Sediment µg/kg
4-Chlorophenyl phenyl ether	10	660
Chrysene	10	660
Dibenz(a,h)anthracene	10	660
Dibenzofuran	10	660
Di-n-butylphthalate	10	ND
1,2-Dichlorobenzene	10	660
1,3-Dichlorobenzene	10	660
1,4-Dichlorobenzene	10	660
3,3'-Dichlorobenzidine	20	1300
2,4-Dichlorophenol	10	660
Diethylphthalate	10	660
2,4-Dimethylphenol	10	660
Dimethyl phthalate	10	660
4,6-Dinitro-2-methylphenol	50	3300
2,4-Dinitrophenol	50	3300
2,4-Dinitrotoluene	10	660
2,6-Dinitrotoluene	10	660
Di-n-octyl phthalate	10	660
Fluoranthene	10	660
Fluorene	10	660
Hexachlorobenzene	10	660
Hexachlorobutadiene	10	660
Hexachlorocyclopentadiene	10	660
Hexachloroethane	10	660
Indeno(1,2,3-cd)pyrene	10	660
Isophorone	10	660
2-Methylnaphthalene	10	660
2-Methylphenol	10	660
3-Methylphenol	10	ND
4-Methylphenol	10	660
Naphthalene	10	660
2-Nitroaniline	50	3300
3-Nitroaniline	50	3300
4-Nitroaniline	20	ND
Nitrobenzene	10	660
2-Nitrophenol	10	660
4-Nitrophenol	50	3300
N-Nitrosodiphenylamine	10	660
N-Nitroso-di-n-propylamine	10	660
Pentachlorophenol	50	3300
Phenanthrene	10	660
Phenol	10	660
Pyrene	10	660
Pyridine	ND	ND
1,2,4-Trichlorobenzene	10	660
2,4,5-Trichlorophenol	10	660

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Semivolatiles	Ground Water µg/L	Low Soil/ Sediment µg/kg
2,4,6-Trichlorophenol	10	660

PQLs listed for soil/sediment are based on wet mass. Normally data is reported on a dry-weight basis, therefore, PQLs will be higher based on the % moisture in each sample. This is based on a 30-g sample and gel permeation chromatography cleanup.

b Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.
ND - Not determined.
NA - Not applicable.

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Table 5 - DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	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

Table 6 - CALIBRATION CHECK COMPOUNDS (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenyl.ami-ne	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

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Table 7 - SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaghthene-d10
Benzyl alcohol	Benzoic acid	Acenaphthene
Bis(2-chloroethyl)ether	Bis(2-chloroethoxy)methane	Acenaphthylene
Bis(2-chloroisopropyl)ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	2,4-Dimethylphenol	Dimethyl phthalate
2-Fluorophenol (surr.)	Hexachlorobutadiene	2,4-Dinitrophenol
Hexachloroethane	Isophorone	2,4-Dinitrotoluene
2-Methylphenol	Naphthalene	2,6-Dinitrotoluene
4-Methylphenol	Nitrobenzene	Fluorene
N-Nitrosodimethylamine	Nitrobenzene-d8 (surr.)	2-Fluorobiphenyl (surr.)
N-Nitroso-di-n-propylamine	2-Nitrophenol	Hexachlorocyclopentadiene
Phenol	1,2,4-Trichlorobenzene	2-Nitroaniline
Phenol-d6 (surr.)		3-Nitroaniline
		4-Nitroaniline
		4-Nitrophenol
		1,2,4,5-Tetrachlorobenzene
		2,4,6-Tribromophenol (surr.)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

Phenanthrene-d10	Chrysene-d12	Perylene-d12
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl)phthalate	Benzo(g,h,i)perylene
Di-n-butyl phthalate	Butylbenzylphthalate	Benzo(a)pyrene
4,6-Dinitro-2-methylphenol	Chrysene	Dibenz(a,h)anthracene
1,2-Diphenylhydrazine	3,3'-Dichlorobenzidine	Di-n-octylphthalate
Fluoranthene	Pyrene	Indeno(1,2,3-cd)pyrene
Hexachlorobenzene	Terphenyl-dl4 (surr.)	
N-Nitrosodiphenylamine		
Pentachlorophenol		
Pentachloronitrobenzene		
Phenacetin		
Phenanthrene		

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Table 8 - QC ACCEPTANCE CRITERIAa
(EPA METHOD 8270C, 1996 Rev. 3)

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p_s (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Anthracene	100	32.0	43.4-118.0	27.133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(ghi)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
Bis(2-chloroethyl)ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl)ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl)phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octylphthalate	100	31.4	18.6-131.8	4-146
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134

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Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for x (µg/L)	Range p, p_s (%)
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation of four recovery measurements, in µg/L.
x = Average recovery for four recovery measurements, in µg/L.
p, p_s = Percent recovery measured.
D = Detected; result must be greater than zero.

a criteria from 40 CFR Part 136 for Method 625. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7.

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Table 9 - ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION
(EPA METHOD 8270C, 1996 Rev. 3)

Parameter	Accuracy, as recovery, X' (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S' (µg/L)
Acenaphthene	0.96C+0.19	0.15x-0.12	0.21x-0.67
Acenaphthylene	0.89C+0.74	0.24x-1.06	0.26x-0.54
Anthracene	0.80C+0.68	0.21x-0.32	0.27x-0.64
Benz(a)anthracene	0.88C-0.60	0.15x+0.93	0.26x-0.21
Benzo(b)fluoranthene	0.93C-1.80	0.22x+0.43	0.29x+0.96
Benzo(k)fluoranthene	0.87C-1.56	0.19x+1.03	0.35x+0.40
Benzo(a)pyrene	0.90C-0.13	0.22x+0.48	0.32x+1.35
Benzo(ghi)perylene	0.98C-0.86	0.29x+2.40	0.51x-0.44
Benzyl butyl phthalate	0.66C-1.68	0.18x+0.94	0.53x+0.92
Bis(2-chloroethyl)ether	0.86C-1.54	0.35x-0.99	0.35x+0.10
Bis(2-chloroethoxy)methane	1.12C-5.04	0.16x+1.34	0.26x+2.01
Bis(2-chloroisopropyl)ether	1.03C-2.31	0.24x+0.28	0.25x+1.04
Bis(2-ethylhexyl)phthalate	0.84C-1.18	0.26x+0.73	0.36x+0.67
4-Bromophenyl phenyl ether	0.91C-1.34	0.13x+0.66	0.16x+0.66
2-Chloronaphthalene	0.89C+0.01	0.07x+0.52	0.13x+0.34
4-Chlorophenyl phenyl ether	0.91C+0.53	0.20x-0.94	0.30x-0.46
Chrysene	0.93C-1.00	0.28x+0.13	0.33x-0.09
Dibenzo(a,h)anthracene	0.88C+4.72	0.30x+8.51	0.59x+0.25
Di-n-butyl phthalate	0.59C+0.71	0.13x+1.16	0.39x+0.60
1,2-Dichlorobenzene	0.80C+0.28	0.20x+0.47	0.24x+0.39
1,3-Dichlorobenzene	0.86C-0.70	0.25x+0.68	0.41x+0.ll
1,4-Dichlorobenzene	0.73C-1.47	0.24x+0.23	0.29x+0.36
3,3-Dichlorobenzidine	1.23C-12.65	0.28x+7.33	0.47x+3.45
Diethyl phthalate	0.43+1.00	0.28x+1.44	0.52x+0.22
Dimethyl phthalate	0.20C+1.03	0.54x+0.19	1.05x-0.92
2,4-Dinitrotoluene	0.92C-4.81	0.12x+1.06	0.21x+1.50
2,6-Dinitrotoluene	1.06C-3.60	0.14x+1.26	0.19x+0.35
Di-n-octylphthalate	0.76C-0.79	0.21x+1.19	0.37x+1.19
Fluoranthene	0.81C+1.10	0.22x-0.73	0.28x-0.60
Fluorene	0.90C-0.00	0.12x+0.26	0.13x+0.61
Hexachlorobenzene	0.74C+0.66	0.18x-0.10	0.43x-0.52
Hexachlorobutadiene	0.71C-1.01	0.19x+0.92	0.26x+0.49
Hexachloroethane	0.73C-0.83	0.17x+0.67	0.17x+0.80
Indeno(1,2,3-cd)pyrene	0.78C-3.10	0.29x+1.46	0.50x-0.44
Isophorone	1.12C+1.41	0.27x+0.77	0.33x+0.26
Naphthalene	0.76C+1.58	0.21x-0.41	0.30x-0.68
Nitrobenzene	1.09C-3.05	0.19x+0.92	0.27x+0.21
N-Nitrosodi-n-propylamine	1.12C-6.22	0.27x+0.68	0.44x+0.47
Phenanthrene	0.87C+0.06	0.12x+0.57	0.15x+0.25
Pyrene	0.84C-0.16	0.16x+0.06	0.15x+0.31
1,2,4-Trichlorobenzene	0.94C-0.79	0.15x+0.85	0.21x+0.39
4-Chloro-3-methylphenol	0.84C+0.35	0.23x+0.75	0.29x+1.31
2-Chlorophenol	0.78C+0.29	0.18x+1.46	0.28x+0.97

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Parameter	Accuracy, as recovery, X' (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S' (µg/L)
2,4-Dichlorophenol	0.87C-0.13	0.15x+1.25	0.21x+1.28
2,4-Dimethylphenol	0.71C+4.41	0.16x+1.21	0.22x+1.31
2,4-Dinitrophenol	0.81C-18.04	0.38x+2.36	0.42x+26.29
2-Methyl-4,6-dinitrophenol	1.04C-28.04	0.10x+42.29	0.26x+23.10
2-Nitrophenol	0.07C-1.15	0.16x+1.94	6.27x+2.60
4-Nitrophenol	0.61C-1.22	0.38x+2.57	0.44x+3.24
Pentachlorophenol	0.93C+1.99	0.24x+3.03	0.30x+4.33
Phenol	0.43C+1.26	0.26x+0.73	0.35x+0.58
2,4,6-Trichlorophenol	0.91C-0.18	0.16x+2.22	0.22x+1.81

X'= Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.

s_r'= Expected single analyst standard deviation of measurements at an average concentration of x, in µg/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of x, in µg/L.

C = True value for the concentration, in µg/L.

x = Average recovery found for measurements of samples containing a concentration of C, in µg/L.

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Table 10
SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES
(EPA CLP SOW OLM 3.0)

		Low/Medium
Surrogate Compound	**Water**	**Soil/Sediment**
Nitrobenzene-d	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
p-Terphenyl-dl4	33-141	18-137
1,2-Dichlorobenzene(advisory)	16-110	20-130
Phenol-d6	10-94	24-113
2-Fluorophenol	21-100	25-121
2,4,6-Tribromophenol	10-123	19-122
2-Chlorophenol-d4(advisory)	33-110	20-130

Table 11
LCS SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES
(EPA CLP SOW OLM 3.0)

	%Recovery	RPD	%Recovery	RPD
Compound	**Water**	**Water**	**Soil**	**Soil**
Phenol	12-110	42	26-90	35
2-Chlorophenol	27-123	40	25-102	50
1,4-Dichlorobenzene	36-97	28	28-104	27
N-Nitroso-di-n-propylamine	41-116	38	41-126	38
1,2,4,-Trichlorobenzene	39-98	28	38-107	23
4-Chloro-3-methylphenol	23-97	42	26-103	33
Acenaphthene	46-118	31	31-137	19
4-Nitrophenol	10-80	50	11-114	50
2,4-Dinitrotoluene	24-96	38	28-89	47
Pentachlorophenol	9-103	50	17-109	47
Pyrene	26-127	31	35-142	36

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Appendix 1 - Weyerhaeuser MDL Study

Matrix: (soil/water) Water
MDL Aliquot: 500 (ul)
Concentrated Extract Volume: 500 (ul)
Injection Volume: 1.0 (ul)
Units: PPB (ug/L)

Date Extracted: 1/9/2004
Date Analyzed: 1/13/2004
Extraction analyst Jim Sanderson
GC/MS analyst Jim Sanderson

Analyte	A	B	C	D	E	F	G
Phenol	4.69	4.86	4.13	4.62	4.45	4.65	4.73
Bis(2-chloroethyl)ether	4.85	4.35	3.82	4.58	4.37	4.41	4.78
2-Chlorophenol	4.46	4.30	3.95	4.40	4.22	4.52	4.45
1,3-Dichlorobenzene	2.74	2.30	2.36	2.76	2.90	2.89	3.29
1,4-Dichlorobenzene	2.88	2.48	2.43	2.75	3.10	2.88	3.18
1,2-Dichlorobenzene	3.13	2.66	2.79	2.97	3.24	3.06	3.58
2-Methylphenol	4.26	4.16	3.77	3.78	4.39	3.94	4.00
2,2'-oxybis(1-chloropropane)	3.68	3.57	3.17	3.51	3.53	3.61	3.77
N-Nitrosodinpropylamine	4.48	4.58	3.99	4.20	4.53	4.27	4.62
4-Methylphenol	4.11	4.28	3.90	3.83	4.46	4.03	3.98
Hexachloroethane	2.20	1.83	1.86	2.14	2.37	2.33	2.70
Nitrobenzene	4.91	4.32	3.85	4.78	4.73	4.63	4.68
Isophorone	4.95	4.75	4.52	4.52	4.80	4.70	4.90
2-Nitrophenol	4.30	4.00	3.72	4.16	4.15	4.21	4.12
2,4-Dimethyphenol	2.28	2.39	2.04	1.53	3.31	1.71	1.60
Bis(2-chloroethoxy)methane	4.29	3.88	3.82	3.97	4.16	4.12	4.27
2,4-Dichlorophenol	4.49	4.50	3.96	4.50	4.56	4.36	4.43
1,2,4-Trichlorobenzene	2.92	2.53	2.63	2.95	3.26	3.03	3.32
Naphthalene	3.98	3.60	3.40	3.95	3.92	3.86	4.09
4-Chloroaniline	5.07	4.35	4.32	4.31	3.52	4.71	4.62
Hexachlorobutadiene	2.03	1.61	1.85	2.09	2.32	2.20	2.79
4-Chloro-3-Methylphenol	4.24	4.15	4.03	4.28	4.62	4.40	4.37
2-Methylnaphthalene	3.84	3.40	3.44	3.75	3.96	3.72	4.16
Hexachlorocyclopentadiene	<1	<1	<1	<1	<1	<1	<1
2,4,6-Trichlorophenol	4.51	4.35	4.17	4.38	4.22	4.20	4.31
2,4,5-Trichlorphenol	4.35	4.06	3.86	3.98	4.41	4.18	4.27
2-Chloronaphthalene	3.97	3.69	3.50	3.93	4.03	3.78	4.20
2-Nitroaniline	5.24	3.91	4.87	4.80	4.21	5.00	4.93
Acenaphthylene	4.24	4.08	3.90	4.33	4.31	4.31	4.36
2,6-Dinitrotoluene	4.88	4.20	4.49	4.52	4.64	4.75	4.66
3-Nitroaniline	4.48	4.39	4.54	4.63	4.59	4.76	4.66
Acenaphthene	4.23	3.92	3.74	4.12	4.23	4.16	4.23
2,4-Dinitrophenol	5.87	5.79	6.37	6.29	6.46	6.56	5.94
Dibenzofuran	4.49	3.96	4.18	4.50	4.47	4.47	4.45
4-Nitrophenol	5.42	4.86	5.56	5.67	5.94	5.68	5.34
2,4-Dinitrotoluene	5.16	4.51	5.28	5.11	5.13	5.13	5.27
Fluorene	4.51	3.85	4.16	4.41	4.58	4.58	4.52
Dimethylphthalate	4.98	4.46	4.61	4.60	4.61	4.64	4.71
Diethylphthalate	4.79	4.36	4.75	4.63	4.70	4.72	4.74
4-Chlorophenyl-phenylether	4.40	3.63	4.05	4.38	4.39	4.41	4.37

Proprietary — Disclosure limited to persons confidentially bound to Weyerhaeuser.

Matrix: (soil/water) Water
MDL Aliquot: 500 (ul)
Concentrated Extract Volume: 500 (ul)
Injection Volume: 1.0 (ul)
Units: PPB (ug/L)

Date Extracted: 1/9/2004
Date Analyzed: 1/13/2004
Extraction analyst Jim Sanderson
GC/MS analyst Jim Sanderson

Analyte	A	B	C	D	E	F	G
4-Nitroaniline	4.96	3.71	4.96	5.13	4.81	5.30	5.14
4,6-Dinitro-2-methylphenol	5.36	5.54	5.78	5.71	5.72	5.89	5.40
N-Nitrosodiphenylamine	4.93	4.70	4.59	4.54	3.49	4.75	5.04
4-Bromophenyl-phenylether	5.09	5.05	4.70	4.86	4.69	5.14	5.01
Hexachlorobenzene	5.13	5.03	4.51	4.70	5.07	4.88	4.98
Pentachlorophenol	5.64	6.04	5.94	6.23	6.50	6.44	6.07
Phenanthrene	5.29	4.94	4.93	5.10	5.24	5.15	5.24
Anthracene	5.01	4.75	4.84	4.98	4.87	5.24	5.01
Di-n-Butylphthalate	5.53	5.36	5.65	5.42	5.61	5.59	5.59
Fluoranthene	4.65	4.22	4.87	4.90	4.93	4.93	4.66
Pyrene	6.02	6.47	5.84	5.58	5.30	5.98	5.90
Butylbenzylphthalate	5.37	5.52	5.35	5.13	4.86	5.31	5.29
Benzo(a)Anthracene	5.38	5.34	5.40	5.30	5.14	5.45	5.47
3,3'-Dichlorobenzidine	3.30	2.87	3.04	2.86	0.51	2.72	3.03
Chrysene	5.56	5.57	5.56	5.50	5.34	5.52	5.80
bis(2-ethylhexyl)Phthalate	5.60	5.53	5.70	5.03	5.07	5.23	5.56
Di-n-octylphthalate	4.88	5.05	5.02	4.76	4.71	4.90	4.81
Benzo(b)fluoranthene	4.79	4.74	4.95	4.54	4.46	4.66	4.66
Benzo(k)fluoranthene	4.85	4.81	4.80	4.89	5.06	4.98	5.38
Benzo(a)pyrene	4.57	4.39	4.45	4.35	4.27	4.52	4.55
Indeno(1,2,3-cd)pyrene	4.54	4.45	4.49	4.48	4.60	4.51	4.47
Dibenzo(a,h)anthracene	4.43	4.43	4.40	4.26	4.41	4.38	4.39
Benzo(g,h,i)perylene	4.81	4.71	4.74	4.62	4.78	4.66	4.78
Carbazole	6.01	5.12	5.70	6.08	5.49	6.17	5.79
Aniline	4.89	4.96	4.70	4.12	1.48	4.95	4.65
Benzyl Alcohol	5.37	5.28	4.81	4.94	5.20	5.12	5.40
Benzoic Acid	4.77	5.11	4.93	5.12	6.07	5.22	4.87
Benzidine	<10	<10	<10	<10	<10	<10	<10
1,2-Diphenylhydrazine	4.99	5.10	4.61	4.82	4.62	4.86	5.06

Proprietary — Disclosure limited to persons confidentially bound to Weyerhaeuser.

Matrix: (soil/water) Water
MDL Aliquot: 500 (ul)
Concentrated Extract Volume: 500 (ul)
Injection Volume: 1.0 (ul)
Units: PPB (ug/L)

Date Extracted: 1/9/2004
Date Analyzed: 1/13/2004
Extraction analyst Jim Sanderson
GC/MS analyst Jim Sanderson

Analyte	MEAN	STDEV	MDL	SPIKE	% Rec
Phenol	4.59	0.237	0.7	5	92
Bis(2-chloroethyl)ether	4.45	0.342	1.1	5	89
2-Chlorophenol	4.33	0.196	0.6	5	87
1,3-Dichlorobenzene	2.75	0.339	1.1	5	55
1,4-Dichlorobenzene	2.81	0.285	0.9	5	56
1,2-Dichlorobenzene	3.06	0.302	1.0	5	61
2-Methylphenol	4.04	0.237	0.7	5	81
2,2'-oxybis(1-chloropropane)	3.55	0.190	0.6	5	71
N-Nitrosodinpropylamine	4.38	0.233	0.7	5	88
4-Methylphenol	4.08	0.221	0.7	5	82
Hexachloroethane	2.20	0.303	1.0	5	44
Nitrobenzene	4.56	0.361	1.1	5	91
Isophorone	4.73	0.169	0.5	5	95
2-Nitrophenol	4.09	0.188	0.6	5	82
2,4-Dimethyphenol	2.12	0.620	1.9	5	42
Bis(2-chloroethoxy)methane	4.07	0.186	0.6	5	81
2,4-Dichlorophenol	4.40	0.204	0.6	5	88
1,2,4-Trichlorobenzene	2.95	0.294	0.9	5	59
Naphthalene	3.83	0.242	0.8	5	77
4-Chloroaniline	4.41	0.480	1.5	5	88
Hexachlorobutadiene	2.13	0.373	1.2	5	43
4-Chloro-3-Methylphenol	4.30	0.190	0.6	5	86
2-Methylnaphthalene	3.75	0.271	0.9	5	75
Hexachlorocyclopentadiene	<1	N.M.	N.M.	5	N.M.
2,4,6-Trichlorophenol	4.31	0.120	0.4	5	86
2,4,5-Trichlorphenol	4.16	0.201	0.6	5	83
2-Chloronaphthalene	3.87	0.233	0.7	5	77
2-Nitroaniline	4.71	0.472	1.5	5	94
Acenaphthylene	4.22	0.168	0.5	5	84
2,6-Dinitrotoluene	4.59	0.218	0.7	5	92
3-Nitroaniline	4.58	0.122	0.4	5	92
Acenaphthene	4.09	0.190	0.6	5	82
2,4-Dinitrophenol	6.18	0.310	1.0	5	124
Dibenzofuran	4.36	0.209	0.7	5	87
4-Nitrophenol	5.50	0.342	1.1	5	110
2,4-Dinitrotoluene	5.08	0.262	0.8	5	102
Fluorene	4.37	0.273	0.9	5	87
Dimethylphthalate	4.66	0.160	0.5	5	93
Diethylphthalate	4.67	0.145	0.5	5	93
4-Chlorophenyl-phenylether	4.23	0.295	0.9	5	85

Matrix: (soil/water) Water
Date Extracted: 1/9/2004

Proprietary — Disclosure limited to persons confidentially bound to Weyerhaeuser.

MDL Aliquot: 500 (ul)
Concentrated Extract Volume: 500 (ul)
Injection Volume: 1.0 (ul)
Units: PPB (ug/L)

Date Analyzed: 1/13/2004
Extraction analyst: Jim Sanderson
GC/MS analyst: Jim Sanderson

Analyte	MEAN	Std Dev	MDL	Amount Spiked	% Recovery
4-Nitroaniline	4.86	0.531	1.7	5	97
4,6-Dinitro-2-methylphenol	5.63	0.199	0.6	5	113
N-Nitrosodiphenylamine	4.58	0.511	1.6	5	92
4-Bromophenyl-phenylether	4.93	0.185	0.6	5	99
Hexachlorobenzene	4.90	0.223	0.7	5	98
Pentachlorophenol	6.12	0.297	0.9	5	122
Phenanthrene	5.13	0.146	0.5	5	103
Anthracene	4.96	0.158	0.5	5	99
Di-n-Butylphthalate	5.54	0.107	0.3	5	111
Fluoranthene	4.74	0.258	0.8	5	95
Pyrene	5.87	0.366	1.2	5	117
Butylbenzylphthalate	5.26	0.211	0.7	5	105
Benzo(a)Anthracene	5.35	0.111	0.3	5	107
3,3'-Dichlorobenzidine	2.62	0.948	3.0	5	52
Chrysene	5.55	0.136	0.4	5	111
bis(2-ethylhexyl)Phthalate	5.39	0.273	0.9	5	108
Di-n-octylphthalate	4.88	0.127	0.4	5	98
Benzo(b)fluoranthene	4.69	0.162	0.5	5	94
Benzo(k)fluoranthene	4.97	0.205	0.6	5	99
Benzo(a)pyrene	4.44	0.112	0.4	5	89
Indeno(1,2,3-cd)pyrene	4.51	0.051	0.2	5	90
Dibenzo(a,h)anthracene	4.39	0.059	0.2	5	88
Benzo(g,h,i)perylene	4.73	0.069	0	5	95
Carbazole	5.77	0.370	1.2	5	115
Aniline	4.25	1.26	3.9	5	85
Benzyl Alcohol	5.16	0.220	0.7	5	103
Benzoic Acid	5.16	0.433	1.4	5	103
Benzidine	<10	N.M.	N.M.	5	N.M.
1,2-Diphenylhydrazine	4.87	0.198	0.6	5	97

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Appendix 2 - TYPICAL SURROGATE AND LCS PERCENT RECOVERIES

PRECISION & ACCURACY
STUDY 8270

Date: 03/07/94
Data: 2SV40304A-2SV40304E
Method: 8270
Analyst: J. Leong
Extractionist: T. Sutherland
Extractionist: M. Luthy
Extraction Date: 2/22/94
Spiked: 50µg/L

Replicate #	% recovery (P) #1	#2	#3	#4	average (X) (% rec.)	Std.Dev. (s)	compliance	limit for s	Range for X (% rec.)	Range for P
PHENOL	53.7	55.5	53.9	58.3	55.4	2.1	Y	22.6	16.6-100.0	5-112
BIS(2-CHLOROETHYL)ETHER	73.9	78.1	78.9	69.2	75.0	4.4	Y	55	42.9-126.0	12-158
2-CHLOROPHENOL	62.4	65.2	61.1	65.1	63.4	2.0	Y	28.7	36.2-120.4	23-134
1,3-DICHLOROBENZENE	60.0	72.6	72.2	62.4	66.8	6.5	Y	41.7	16.7-153.9	d-172
1,4-DICHLOROBENZENE	61.8	72.0	72.0	62.9	67.1	5.6	Y	32.1	37.3-105.7	20-124
1,2-DICHLOROBENZENE	62.5	70.4	69.4	62.3	66.1	4.3	Y	30.9	48.6-112.0	32-129
2-METHYLPHENOL	73.8	77.5	74.2	74.4	75.0	1.7		NA		
2,2'-OXYBIS(1-CHLOROPROPANE)	83.1	86.7	84.8	74.7	82.3	5.3	Y	46.3	62.8-138.6	36-166
4-METHYLPHENOL	75.8	74.7	76.2	77.1	76.0	1.0		NA		
N-NITROSO-DI-N-PROPYLAMINE	79.4	81.5	81.9	73.2	79.0	4.0	Y	55.4	13.6-197.9	d-230

HEXACHLOROETHANE	57.3	75.6	75.4	65.6	68.5	8.8	Y	24.5	55.2-100.0	40-113
NITROBENZENE	73.6	78.0	75.8	67.2	73.6	4.7	Y	39.3	54.3-157.6	35-180
ISOPHORONE	79.7	82.3	83.4	73.5	79.7	4.4	Y	63.3	46.6-180.2	21-196
2-NITROPHENOL	78.4	82.7	78.4	84.7	81.0	3.1	Y	35.2	45.0-166.7	29-182
2,4-DIMETHYLPHENOL	59.9	52.4	63.8	50.1	56.5	6.4	Y	26.1	41.8-109.0	32-119
BIS(2-CHLOROETHOXY)METHANE	82.3	84.5	85.0	75.8	81.9	4.2	Y	34.5	49.2-164.7	33-184
2,4-DICHLOROPHENOL	72.3	76.5	72.0	77.6	74.6	2.9	Y	26.4	52.5-121.7	39-135
1,2,4-TRICHLOROBENZENE	70.0	81.0	80.5	71.0	75.6	5.9	Y	28.1	57.3-129.2	44-142
NAPHTHALENE	73.8	79.2	79.9	69.6	75.6	4.9	Y	30.1	35.6-119.6	21-133
4-CHLOROANILINE	78.3	81.3	83.7	72.9	79.0	4.7		NA		
HEXACHLOROBUTADIENE	66.0	82.6	81.9	71.3	75.4	8.2	Y	26.3	37.8-102.2	24-116
4-CHLORO-3-METHYLPHENOL	76.5	78.7	77.9	77.2	77.5	0.9	Y	37.2	40.8-127.9	22-147
2-METHYLNAPHTHALENE	77.6	80.3	81.9	74.7	78.6	3.2		NA		
HEXACHLOROCYCLOPENTADIENE	3.0	2.4	4.1	3.9	3.4	0.8		NA		
2,4,6-TRICHLOROPHENOL	77.8	81.4	73.9	83.3	79.1	4.1	Y	31.7	52.4-129.2	37-144
2,4,5-TRICHLOROPHENOL	76.7	76.0	70.8	74.9	74.6	2.7		NA		
2-CHLORONAPHTHALENE	80.2	84.1	84.9	77.4	81.6	3.5	Y	13.0	64.5-113.5	60-118
2-NITROANILINE	78.4	81.3	83.5	74.2	79.3	4.0		NA		
DIMETHYLPHTHALATE	36.4	68.3	71.9	67.5	61.0	16.5	Y	23.2	d-100.0	d-112
ACENAPHTHYLENE	76.4	79.3	81.0	73.7	77.6	3.2	Y	40.2	53.5-126.0	33-145

2,6-DINITROTOLUENE	80.9	85.9	90.2	80.7	84.4	4.6	Y	29.6	68.1-136.7	50-158
3-NITROANILINE	88.9	92.9	95.6	88.4	91.5	3.4		NA		
ACENAPHTHENE	77.4	80.3	80.3	73.0	77.8	3.4	Y	27.6	60.1-132.3	47-145
2,4-DINITROPHENOL	81.1	86.3	79.6	92.0	84.7	5.6	Y	49.8	d-172.9	d-191
4-NITROPHENOL	73.8	78.2	67.0	74.9	73.5	4.7	Y	47.2	13.0-106.5	d-132
DIBENZOFURAN	79.5	83.0	85.0	76.6	81.0	3.7		NA		
2,4-DINITROTOLUENE	82.1	83.8	87.7	80.7	83.6	3.0	Y	21.8	47.5-126.9	39-139
DIETHYLPHTHALATE	74.5	87.6	92.2	86.0	85.1	7.5	Y	26.5	d-100.0	d-114
4-CHLOROPHENYLPHENYLETHER	88.9	93.1	95.6	87.7	91.3	3.7	Y	33.4	38.4-144.7	25-158
FLUORENE	80.8	81.8	85.8	78.6	81.7	3.0	Y	20.7	71.6-108.4	59-121
4-NITROANILINE	86.6	96.3	98.2	92.5	93.4	5.1		NA		
4,6-DINITRO-2-METHYLPHENOL	73.9	76.9	72.1	78.3	75.3	2.8	Y	93.2	53.0-100.0	d-181
N-NITROSODIPHENYLAMINE	78.5	96.4	98.6	90.5	91.0	9.0		NA		
4-BROMOPHENYLPHENYLETHER	94.6	98.3	101.3	91.8	96.5	4.2	Y	23	64.9-114.4	53-127
HEXACHLOROBENZENE	90.2	94.9	98.3	88.7	93.0	4.4	Y	24.9	7.8-141.5	d-152
PENTACHLOROPHENOL	86.9	92.0	82.1	92.7	88.5	4.9	Y	48.9	38.1-151.8	14-176
PHENANTHRENE	82.1	83.9	86.2	77.7	82.5	3.6	Y	20.6	65.2-108.7	54-120
ANTHRACENE	84.5	86.5	90.0	80.4	85.4	4.0	Y	32	43.4-118.0	27-133
CARBAZOLE	118.8	127.5	133.2	123.5	125.8	6.1		NA		
DI-N-BUTYLPHTHALATE	87.2	92.0	96.7	86.1	90.5	4.9	Y	16.7	8.4-111.0	1-118
FLUORANTHENE	82.0	85.2	88.1	79.5	83.7	3.8	Y	32.8	42.9-121.3	26-137
PYRENE	86.1	91.1	87.1	82.1	86.6	3.7	Y	25.2	69.6-100.0	52-115

BUTYLBENZYLYPHTHALATE	88.0	95.8	91.6	84.7	90.0	4.7	Y	23.4	d-139-9	d-152
3,3'-DICHLOROBENZIDENE	129.4	150.0	146.8	132.3	139.6	10.3	Y	71.4	8.2-212.5	d-262
BENZO(A)ANTHRACENE	83.2	93.6	84.3	82.2	85.8	5.3	Y	27.6	41.8-133.0	33-143
CHRYSENE	83.1	88.7	88.2	80.9	85.2	3.8	Y	48.3	44.1-139.9	17-168
BIS(2-ETHYLHEXYL)PHTHLATE	88.0	94.7	90.3	83.0	89.0	4.9	Y	41.1	28.9-136.8	8-158
DIOCTYLPHTHALATE	81.9	83.8	89.6	79.0	83.5	4.5	Y	31.4	18.6-131.8	4-146
BENZO(B)FLUORANTHENE	79.9	83.7	85.6	77.1	81.6	3.8	Y	38.8	42.0-140.4	24-159
BENZO(K)FLUORANTHENE	73.8	70.3	79.0	72.0	73.8	3.7	Y	32.3	25.2-145.7	11-162
BENZO(A)PYRENE	79.7	81.4	85.2	78.6	81.2	2.9	Y	39	31.7-148.0	17-163
INDENO(1,2,3-CD)PYRENE	84.3	92.0	88.8	82.4	86.9	4.4	Y	44.6	d-150.9	d-171
DIBENZ(A,H)ANTHRACENE	86.0	93.5	94.7	86.7	90.2	4.5	Y	70	d-199.7	d-227
BENZO(G,H,I)PERYLENE	80.6	83.5	88.2	80.1	83.1	3.7	Y	58.9	d-195.0	d-219

Appendix 3 - METHOD MODIFICATIONS FOR MARINE SEDIMENT "PSEP."

INTRODUCTION – Analytical Method AM E-8270 requires modifications to address specific low reporting limits and reporting units.

MODIFICATIONS – The following modifications are required:

I. EXTRACTION PROCEDURE RECOMMENDATION
II. ORGANIC CARBON
III. REPORTING LIMITS
IV. SAMPLE AMOUNT
V. EXTRACTION MODIFICATIONS
VI. CALIBRATION STANDARDS
VII. REPORTING UNITS

I. EXTRACTION PROCEDURE RECOMMENDATION

The recommended extraction method for marine sediments is soxhlet extraction, SOP AC G-3540. Ultrasonic extraction, SOP AC G-3350, is an acceptable alternative; however comparable recoveries to SOP AC G-3540 must be confirmed.

II. ORGANIC CARBON

A. Numerical reporting values for organic analytes will be normalized based on organic carbon.

B. The analysis for total organic carbon, as well as the percent moisture will be performed by Group 4 (Conventional Group).

C. Total organic carbon and percent moisture data will be used to calculate sample size.

III. REPORTING LIMITS

The reporting limits for marine sediment is analyte specific. The reporting limits are detailed in Table A1. However, a 380-µg/kg organic carbon reporting limit will be used as the basis for determining sample size, final sample extract volume, and calibration standard concentration.

IV. SAMPLE AMOUNT

Use the following formula to determine sample size:

$$\text{Sample size (g)} = \frac{(LS)(F_v)(2)}{(380)(D)(TOC)}$$

Where:

LS = Concentration of the lowest calibration standard in ug/mL. For marine sediment the low calibration standard concentration is 1.0 ug/mL.

F_V = Final volume of the sample extract. For marine sediment the final extract volume is 250 microliters.

The value of 2 is used due to GPC loop loss.

The value of 380 is the resultant analyte concentration in PPB, OC normalized (specified reporting limit).

D = (100 - % moisture in sample)/100.

TOC = percent total organic carbon in the dry weight sample (1 percent TOC = 0.01).

V. EXTRACTION MODIFICATIONS

If the calculated sample size is greater than 60 g the following extraction modifications will need to be made:

A. Weigh the sample into two equal portions. Spike each portion with one half of the surrogate solution, and/or spiking solution. For example, suppose the total sample size is 150 g and one mL of spiking solution is required. The procedure modification would require two sample portions weighing 75 g each. 500 µL of the spiking solution would be added to each sample portion.

B. Combine the sample fraction extracts prior to sample concentration.

C. After GPC cleanup, concentrate the sample extract to 200 µL. Transfer the sample extract to an amber GC vial containing a 300-µL GC vial insert (or equivalent insert). Rinse the concentration tube with small amounts of methylene chloride and combine with the extract in the insert. Continue until a final extract volume of 250 µL is achieved.

VI. CALIBRATION STANDARDS

Include an initial calibration standard at a concentration of 1.0 µg/mL. Verify the curves are valid for the following analytes: 1,2-dichlorobenzene, 1,4-dichlorobenzene, 1,2,4-trichlorobenzene, hexachlorobenzene.

VII. REPORTING UNITS

Report the analyte concentration in mg/kg TOC. Include the concentration for LPAH, HPAH compounds and total benzofluoranthenes. The LPAH criterion represents the sum of the following "low molecular polynuclear aromatic hydrocarbon" compounds: naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, and anthracene. The HPAH criterion represents the sum of the following "high molecular polynuclear aromatic hydrocarbon" compounds: fluoranthene, pyrene, benz(a)anthracene, chrysene, total benzofluoranthenes, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenz(a,h)anthracene, and benzo(g,h,i)perylene. The total benzofluoranthenes criterion represents the sum of the concentrations of "B," "J," and "K" isomers.

Table A1. Reporting limits for marine sediment.

Chemical Parameter	mg/kg Organic Carbon
LPAH	370
Naphthalene	99
Acenaphthylene	66
Acenaphthene	16
Fluorene	23
Phenanthrene	100
Anthracene	220
2-Methylnaphthalene	38

Chemical Parameter	mg/kg Organic Carbon
Bis(2-ethylhexyl) Phthalate	47
Di-n-octyl Phthalate	58
Dibenzofuran	15
Hexachlorobutadiene	3.9
N-Nitrosodiphenylamine	11

	µg/kg Dry

Chemical Parameter	mg/kg Organic Carbon
HPAH	960
Fluoranthene	160
Pyrene	1000
Benz(a)anthracene	110
Chrysene	110
Total Benzofluoranthene	230
Benzo(a)pyrene	99
Indeno(1,2,3-cd)pyrene	34
Dibenz(a,h)anthracene	12
Benzo(g,h,i)perylene	31
1,2-Dichlorobenzene	2.3
1,4-Dichlorobenzene	3.1
1,2,4-Trichlorobenzene	0.81
Hexachlorobenzene	0.38
Dimethyl Phthalate	53
Diethyl Phthalate	61
Di-n-butyl Phthalate	220
Butyl Benzyl Phthalate	4.9

Chemical Parameter	mg/kg Organic Carbon
Chemical Parameter	**Weight(PPB)**
Phenol	420
2-Methylphenol	63
4-Methylphenol	670
2,4-Dimethyl Phenol	29
Pentachlorophenol	360
Benzyl Alcohol	57
Benzoic Acid	650

W-16

Total Solids in Solid and Semi-solid Samples, Determination of

Process Owner (TS/PM/OM/LM)

Dennis Catalano

Assignment

Reviewed May 5, 2006 by Christine Devine
Reviewed August 19, 2008 by Christine Devine

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1.0 SCOPE

1.1 This procedure measures the amount of solids (or moisture) present in soil, sediment, sludge-type materials, pulp, wood, hogfuel, and ash when they are dried at 105 °C.

1.2 This procedure is equivalent to Standard Method 2540G for the determination of percent total solids in these types of samples. This method meets the criteria for all the SL-OD-X codes in LIMS.

1.3 This replaces method AM 1-TS-CLP.

2.0 SUMMARY OF METHOD

A weighed portion of the sample is dried in an oven at 105 °C to determine solids remaining or loss of moisture. The test generally assumes, for all practical purposes, a constant mass is achieved after 12 hr, even though oils, caustics, etc. may prevent such in reality.

3.0 INTERFERENCES

Samples that are oily usually have oil remaining after drying which is considered part of the solids even though it is liquid. If the sample contains large amounts of oil, a constant mass may never be obtained. Volatile organics and ammonium carbonate losses will impart a negative error. Large pieces of 'dry' material, such as large (non-homogeneous) rocks, may distort the actual moisture value. This can distort dulplicate values.

4.0 ESTIMATE OF ANALYTICAL or TESTING TIME

This test takes 15 min per sample.

5.0 AMOUNT OF SAMPLE REQUIRED

A minimum of 5 g of sample is necessary (10 g if a duplicate is to be performed). Some samples such as hogfuel, or soil with large rocks, would require a much larger sample to be representative and adjustments must be made accordingly.

6.0 SAMPLING, SAMPLE HANDLING, AND PRESERVATION

6.1 Refrigerate most solid samples at 4 °C (± 2 °C) upon receipt until analysis. Samples that are dry when received, such as ash or pulp sheets, should not be refrigerated since they could pick up moisture. Although there is no required holding time for this test, it is

recommended that it be performed near the time that aliquots are removed for other tests since the moisture content may change over time.

6.2 Samples, especially when very wet, need to be sealed just after sampling to prevent changing moisture content

7.0 EQUIPMENT REQUIRED

7.1 Oven capable of maintaining 104 °C (± 1 °C)

7.2 Balance with a capable of weighing masses stipulated below to the nearest 0.001 g.

8.0 PROCEDURE

8.1 Oven-dry a suitable container (beaker, crucible, or weigh bottle) for 1 hr, and cool in a desiccator for 1 hr. If samples are corrosive in nature, a beaker or porcelain crucible should be used. If samples contain materials that may prevent a true constant mass from being determined, such as very caustic samples or oily samples, note such on the data sheet. Aluminum pans may be used for non-corrosive samples for this analysis but they do not require this pretreatment step.

NOTE: Length of cooling time should be the same for the tare and for after sample is dried.

NOTE: Beakers and crucibles should not be more that 25-30 g of mass since it is difficult to get accurate weighings. Requirements for using Pt crucibles (such as for FBA analysis) or larger quantities (such as for highly inhomogeneous samples) are recognized needs for exceptions. Large Teflon dishes are used for hogfuel.

8.2 Weigh and record the tare mass to the nearest 0.001 g.

8.3 Mix the sample to achieve homogeneity. Place a 5 -10 g subsample in the pan and record mass of the sample plus the tare to the nearest 0.001 g.

CAUTION: Weigh samples quickly. Do not let the 'wet' sample sit around after aliquoting. This can result in a loss of water and affect the accuracy of the test.

NOTE: For samples that are difficult to homogenize, use a larger aliquot and an appropriately larger vessel.

8.4 Place the pan in an oven maintained at a temperature of 104 °C (± 1 °C) for 12-24 hr, but no longer than 24 hr. If dried less than 12 hr, it must be documented that a constant mass was attained.

NOTE: Drying time is defined as the elapsed time in the oven; thus raw data must record time in and out of the oven to document the 12 hr drying time minimum. If it is necessary to demonstrate the attainment of constant mass, data must be recorded for a minimum of two repetitive weigh/dry/desiccate/weigh cycles with a minimum of 1-hr drying time in each cycle. Constant mass would be defined as a loss of no greater than 0.05 g, or 4 %, whichever is less, between cycle starting mass and final mass of the last cycle.

8.5 Cool the sample in a desiccator. (See note under 8.1.)

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8.6 Weigh the pan with the dried sample to the nearest 0.001 g.

9.0 QUALITY ASSURANCE

A duplicate sample is performed with each batch, or one every 20 samples, whichever is more frequent.

10.0 REPORT

11.1 Calculations:

$$\%\ \text{Total Solids} = \frac{(A - B)}{C} \times 100$$

where: A = gross dry mass of sample plus the pan (in grams)
B = tare mass of the pan (in grams)
C = as-received sample mass (in grams)

11.2 Report % Total Solids to the nearest 0.1 % as-received basis.

11.3 Precision and accuracy are not applicable to this procedure. There is no reference material to make an appropriate determination of accuracy. Precision varies among samples and depends upon the homogeneity.

11.4 Detection limit is not applicable to this procedure. Lower limit will vary according to the overall mass used in the procedure and the relative ability of the balance to weigh mass.

12.0 KEY WORDS

105, AM 1-TS-CLP, AM S-2540G, moisture, pulp, sediment, sludge, soil, solids, total solids, wood

13.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 2540G.

14.0 REVISION HISTORY

Removed stipulation in 8.1 for aluminum pans and made their use optional in cases when the samples are not corrosive to the pans. Changed 'drying' to 'cooling' in 8.1 note.
May 5, 2006-Changed owner to Dennis Catalano. Fixed typos.

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W-17

Procedure for Florisil Clean Up of Organic Extracts

Process Owner (TS/PM/OM/LM)	
Randy Eatherton	

Reviewed June 20, 2003 by Christine Devine
Reviewed September 11, 2007 by Christine Devine

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1.0 SCOPE

Florisil is magnesium silicate. This procedure is written only as a cleanup by absorption for pesticides (AM E-8080, AM E-8081 and AM E-8082) and chlorinated hydrocarbons (method AM E-8121) and is applicable to both sediment/soil and water extracts. Any other compounds must be demonstrated to work with this method (an example would be herbicides and phenolic compounds, which would have to be derivatized in order not to be absorbed permanently onto the Florisil).

1.2 The Florisil absorbs polar compounds. The more polar the compound, the more difficult it is to displace it from the Florisil with solvents. Non-polar compounds pass through the Florisil cartridge without being absorbed (this is why hexane is used to elute PCBs, which are nonpolar). Phenolic compounds are an example of polar compounds that are not desorbed from the Florisil with a polar solvent such as methylene chloride. The organochlorine pesticides are slightly polar, and the desorbing of these compounds has been determined to be eluted with a slightly polar solvent (9 + 1 v/v hexane/acetone). The purpose of the Florisil clean up is to remove highly polar compounds that would interfere with GC analysis.

1.3 This procedure follows the EPA's SW-846 method 3620.

2.0 SUMMARY OF PROCEDURE

2.1 Florisil clean up is accomplished using pre-packed cartridges that have been calibrated per lot before using. The cartridges are 0.5 or 1.0 gram of 40-µm particles of Florisil in either polypropylene (0.5 gram) or glass (1.0 gram) syringe bodies. The glass body is expensive and used exclusively for low level water analysis (1-2 mL final volume on 1-L extracts) and other low level work, such as Puget Sound Protocol soil extractions (1-mL final volume on 75-150-g sediment extracts) and pulp samples (1-mL final volume for 10-g extracts).

2.2 The cartridges are pre-cleaned with hexane. 1 mL of extract is added to the cartridge and eluted with 10 mL of 90 +10 Hexane/Acetone. A vacuum manifold is required to obtain reproducible results. The eluate is further concentrated with nitrogen to 1-mL final volume.

3.0 INTERFERENCES

Analytical interferences may be caused by contaminants in solvent, reagents, glassware, and other sample processing hardware. All of these materials must be demonstrated to be free of interferences by analyzing laboratory reagent blanks. A laboratory blank per batch must demonstrate interferences/analyte hits below the method detection limit before this method is performed on samples.

4.0 ESTIMATE OF ANALYTICAL TIME

Batch sizes are 1 to 12 samples. The preparation time is ½ hour. The elapsed time is usually 2-3 hours (due to gentle concentration of the eluate).

5.0 AMOUNT OF SAMPLE REQUIRED

The amount of sample is not important. The sample extract must be at a final volume of 1.0 mL in order to be added to the Florisil cartridge.

6.0 SAMPLING, SAMPLE HANDLING, AND PRESERVATION

Extracts are allowed to warm to room temperature before performing Florisil clean up.

7.0 EQUIPMENT REQUIRED

Vacuum manifold - Consisting of a glass vacuum basin, collection rack, Teflon manifold outfitting with Teflon needles and on/off valves for each station. A vacuum gauge is on the vacuum inlet in the base. The system is connected to the house vacuum and has a bleed valve connected between the manifold and the vacuum supply. The samples are collected in 15-mL centrifuge tubes.

8.0 REAGENTS REQUIRED

8.1 Florisil cartridges - 40-µm particles, 60-nm (60-Ă) pores. The cartridges consist of: 6-mL serological grade polypropylene tubes with 0.500 g of Florisil held between two polyethylene frits; and 10-mL glass tubes with 1.00 g of Florisil held between two glass frits.

8.2 Eluting solvents - hexane and acetone - pesticide quality. For organochlorine pesticide analysis (AM E-8080 and AM E-8081), the eluting solvent is 90 ± 1 mL hexane +10 ± 1 mL acetone. For Aroclor analysis (AM E-8082), the eluting solvent is hexane only.

Acetone, [67-64-1]

DANGER: Acetone is highly flammable. It has a flash point of -20 °C (-4 °F) and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity).
Avoid breathing vapors. Exposure can cause coughing, dullness, headache, dizziness, nausea, irritation to the eyes and respiratory tract, narcosis, and unconsciousness. Higher concentrations may cause damage to kidneys, liver, and central nervous system (depression). Chronic exposure during pregnancy may be harmful.
Avoid skin contact. Acetone is absorbed through the skin. Prolonged or repeated skin contact can cause severe irritation and dermatitis, because of the defatting action on skin. May cause redness, pain, drying and cracking of the skin. Toxicity of alcohol, and halogentated hydrocarbons may be increased.
Acetone can react vigorously with oxidizing materials. Avoid strong acids, strong alkalis, and halogens and halogen compound.
ACGIH TWA: 500 ppm, 1,188 mg/m^3

Hexane [110-54-3]

DANGER: All manipulations involving hexane must be performed within a fume hood. Nitrile gloves are recommended. n-Hexane is highly flammable. It has a flash point of -23 °C (-9 °F), has explosive limits in air in the range of 1 - 7 %, and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity). n-Hexane can react vigorously with oxidizing materials.

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Avoid breathing vapors. Exposure can cause dizziness, numbness of extremities, and intoxication. n-Hexane is a central nervous system depressant and neurotoxin. Acute exposure causes irritation, narcosis, and gastrointestinal tract irritation. Chronic inhalation causes peripheral neuropathy can have neurotoxic effects.
Avoid skin contact. n-Hexane is absorbed through the skin. Prolonged or repeated skin contact can cause irritation and dermatitis, through defatting of skin.
No PEL given.

8.3 Florisil cartridge phenol check solution - 2,4,5-Trichlorophenol in acetone at a concentration of 100 mg/L. This standard is purchased from either Ultra scientific or Accustandards.

8.4 Florisil cartridge pesticide check solution: The following solution containing the analytes in hexane is prepared (this is the mid-point calibration solution of INDA and INDB combined). These standards are purchased from either Ultra scientific or Accustandards. The standards are combined and gently concentrated on the nitrogen concentrator to a 1.0 ± 0.05 mL final volume.

Analyte	µg/mL	Analyte	µg/mL
alpha BHC	0.20	Endrin Ketone	0.40
Beta BHC	0.20	Endrin Aldehyde	0.40
Delta BHC	0.20	4,4' DDE	0.40
Gamma BHC	0.20	4,4' DDD	0.40
Aldrin	0.20	4,4' DDT	0.40
Heptachlor	0.20	Gamma Chlordane	0.20
Heptachlor Epoxide	0.20	Alpha Chlordane	0.20
Endrin	0.40	Methoxychlor	2.0
Dieldrin	0.40	Tetrachloroxylene	0.40
Endosulfan I	0.20	Decachlorobiphenyl	0.40
Endosulfan II	0.40		

9.0 PROCEDURE

9.1 Cartridge set-up and conditioning.

9.1.1 Place 15-mL centrifuge tubes in the manifold rack and place the top of the manifold on the system. Arrange the cartridges on the manifold in the closed-valve position.

9.1.2 Turn on the vacuum and set the vacuum needle valve to 50 (± 12) mm [2 (± 1) inch] of Hg. This is the only way of controlling the flow rate on the cartridges.

9.1.3 Add a full cartridge volume of hexane + acetone solution to each cartridge. Open the valve and allow enough hexane + acetone (8.2), to elute through the cartridges to remove air bubbles. Close the valve and allow the entire sorbent bed to soak for at least 5 min (but do not allow to dry). There must be enough solvent to keep the bed moist before the cartridges are further used. If the bed becomes dry, this step must be repeated.

9.1.4 Open the cartridge valve. Continue to add hexane + acetone (8.2), until you have conditioned the cartridges by adding 3 cartridge volumes of hexane/acetone to the polypropylene cartridges and 2 cartridge volumes of the hexane + acetone to the glass cartridges. Close the cartridge valve when there is still solvent above the sorbent bed (the meniscus is just visible). Do not allow the cartridge to go dry. If the cartridge goes dry, repeat the conditioning step.

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9.1.5 Remove the centrifuge tubes and solvent. Empty the centrifuge tubes and rinse them with hexane and invert them on a test tube rack. Allow the hexane rinse to drain. Replace the centrifuge tubes in the manifold system.

9.2 Handling sample extracts

9.2.1 Reduce the sample extract volume to 10 ± 0.1 mL (for extracts via AC G-8080, AC G-8081, AC G-8082), 2 ± 0.05 mL (for the low level water analysis), or 1 ± 0.05 mL (for Puget Sound Protocol) prior to clean up. Split off 1.0 ± 0.05 mL for the first two types of analyses with a pre-rinsed 1-mL disposable pipet. The whole sample is cleaned up for Puget Sound Protocol.

9.2.2 Transfer the extract to the cartridge. Open the cartridge valve to allow the extract to pass through the cartridge at 2 ± 1 mL/min. This rate is adjusted with the vacuum needle valve.

9.2.3 When the entire extract has passed through the cartridge, but before the cartridge becomes dry, rinse the sample vial with an addition 0.5 ± 0.1 mL of hexane + acetone (8.2), and add the rinse to the cartridge to complete the quantitative transfer. If the extracts are from AC G-8082, then use hexane as the eluate.

9.2.4 Open the cartridge valve and allow the extract to elute onto the cartridge. Turn off the valve and allow the extract to soak on the Florisil for 5 - 10 min (do not let the top of the bed to dry).

9.2.5 Add a full cartridge volume of hexane + acetone, (in the case of AC G-8082, use hexane only) and open the cartridge valve. Before the cartridge goes dry, continue adding hexane + acetone until 10 ± 0.5 mL of eluate has been collected. Turn off the cartridge valve.

9.2.6 Repeat this until all of the cartridges have been spent. Turn off the vacuum and remove the centrifuge tubes, making sure the sample labels are transferred for the extract vial.

9.2.7 Concentrate the eluate to 1.0 ± 0.05-mL final volume. The sample is transferred into a clear GC vial and capped. It is stored in the refrigerator until ready for analysis. If sulfur is suspected to be present in the sample, mercury clean up (AC G-3660) is applied.

10.0 QUALITY ASSURANCE

The analyst must demonstrate that the compounds of interest are being quantitatively recovered by the clean-up technique before the clean up is applied to samples. The calibration mix, a solvent blank, and the trichlorophenol mix are each passed through Florisil cleanup and analyzed in order to characterize each Florisil batch. For the florisil lot to be acceptable, the recovery of the organochlorine pesticides must be 80 - 110 %, the recovery of trichlorophenol must be less than 5 %, and there must be no peaks interfering with the target compounds in the blank.

11.0 KEY WORDS

3620, aroclors, AC G-3620, clean up, EPA, Florisil, non-polar, organochlorine, PCB, pesticide, polar, SW-846

12.0 REFERENCES

12.1 “Test Methods for Evaluating Solid Waste SW846,” Volume 1B: Laboratory Manual Physical/Chemical Methods, Revision 2, January 1995, 3620B

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12.2 "Statement of Work for Organic Analysis, Multi-media, Multi-concentration," Document Number OLM01.0.

13.0 REVISION HISTORY

13.1 4/10/04 – Changed SOP code from AP E-3620.

13.2 9/11/07 – Updated codes for referenced SOPs and updated safety information.

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W-18

Gel Permeation Clean-up

Process Owner (TS/PM/OM/LM) **Randy Eatherton**	
Assignment	Reviewed November 3, 2005 by Christine Devine Reviewed August 19, 2008 by Christine Devine
Electronic only unless colored "Controlled" stamp is present	

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1.0 SCOPE

1.1 Gel-permeation chromatography (GPC) is a size-exclusion clean up using methylene chloride and a hydrophobic gel (SX-3 Bio Beads) via a low-pressure liquid chromatography system. The packing gel is porous and is characterized by the range/uniformity (exclusion range) of that pore size - where the exclusion range must be larger than the molecular size of the molecules to be separated.

1.2 GPC may be required for soil/sediment/tissue samples for the elimination from the sample of lipids, polymers, copolymers, proteins, natural resins and polymers, steroids and oils.

1.3 The applications of GPC for analytes in methods AM E-8270 and AM E-8080 are valid.

1.4 This procedure follows EPA 3640A.

2.0 SUMMARY OF PROCEDURE

GPC is accomplished using an automated low-pressure liquid chromatography system that has been calibrated before using. Minimal calibration consists of ensuring a maximum resolution for a calibration mix (8080 and 8270) and establishing where corn oil elutes. This corn oil is used as a generic surrogate for oil and establishes where the sample will be collected. An additional calibration mix of pesticides and PCBs is required for method AM E-8080. Before loading samples, the sample tubes and transfer lines are pre-rinsed with 10 mL of solvent.

10 mL of the sample extract in methylene chloride is pre-loaded in glass tubes. The sample is loaded onto the column and eluted through the column with methylene chloride at 5-6 mL/minute flow rate for approximately 50 minutes. The corn oil elution (determined from the calibration) is used as the indicator for where the eluant will be collected. The eluant is concentrated by KD and nitrogen evaporation to a final volume determined by the analytical method.

3.0 INTERFERENCES

Analytical interferences may be caused by contaminants in solvent, reagents, glassware, and other sample processing hardware. All of these materials must be demonstrated to be free of interferences by analyzing laboratory reagent blanks. The GPC is calibrated on a weekly basis. The blank must demonstrate interferences/analyte hits are below the method detection limit* before this method is performed on samples. The methylene chloride must be fresh; stale methylene chloride becomes acidic and it has been demonstrated in the lab this will degrade some of the 8270 surrogates.

*** See SOP for Detection and Quantitation Limits for exceptions to this.**

4.0 ESTIMATE OF ANALYTICAL TIME

4.1 Single sample. A single sample may take 60 to 90 minutes to filter and collect the extract, and wash the GPC system.

4.2 Economical batch size is 6 – 15 samples.

5.0 AMOUNT OF SAMPLE REQUIRED

The desired extract volume is 10 ± 0.2 mL, although a minimum volume of only 8 mL is needed to perform the analysis. The volume must be recorded whenever it is outside the 10 ± 0.2 mL ranges.

6.0 SAMPLING, SAMPLE HANDLING AND PRESERVATION

The sample extract should be refrigerated in a capped, graduated centrifuge tube prior to GPC analysis. Extracts are allowed to warm to room temperature before loading onto the GPC for clean up.

7.0 EQUIPMENT REQUIRED

7.1 Gel-permeation chromatography system - GPC Autoprep Model 1000, Analytical Biochemical Laboratories, Inc.

7.2 Chromatographic column - 700 mm x 25-mm ID glass column. Flow is upward.

7.3 Bio Beads (S-X3) - 200-400 mesh, 70 g. The quality of Bio Beads may vary from lot to lot because of excessive fines. The backpressure should be within 5 - 10 psi.

7.4 Ultraviolet detector - Fixed wavelength (254 nm) with a semi-prep flow-through cell.

7.5 Soltec Z10 integrator/strip chart recorder.

7.6 Glassware and disposables

7.6.1 Syringes - 24 glass 10-mL syringes with luerlock fittings.

7.6.2 Disposable filter disk - PTFE 25-mm diameter, 0.45 µm and 1.0 µm.

7.6.3 GPC sample tubes - 24 threaded amber tubes for loading of the samples.

7.6.4 Erlenmeyer Flasks - 24 125-mL Erlenmeyer flasks for sample collection.

8.0 REAGENTS REQUIRED

8.1 Methylene Chloride - B & J Pesticide quality or equivalent.

DANGER:

All manipulations involving methylene chloride must be performed within a fume hood or a sealed system to prevent venting into the laboratory. It is non-flammable.
Avoid breathing vapors. Exposure can cause anesthetic or narcotic effects, light-headedness, nausea, vomiting and headache. Excessive exposure may cause irritation to upper respiratory tract.

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Unconsciousness and death may result from extreme cases of over exposure. High levels may also cause cardiac arrhythmias (irregular heartbeats). Breathing vapors can elevate carboxyhemoglobin levels in the cardiovascular system thereby impairing the blood's ability to transport oxygen. Persons who smoke tobacco products will experience an intensified elevation of carboxyhemoglobin levels. Observations in animals include liver and kidney effects.
Delayed effects: exposure may aggravate symptoms of angina (chest pains). Liver and kidney damage may occur.
Avoid skin contact. Prolonged or repeated exposure may cause skin irritation, even a burn. Repeated contact may cause drying or flaking of skin. Can cause irritation and dermatitis.
Cancer information: Suspect carcinogen. Causes cancer in mice and benign tumors in rats.
Avoid direct sunlight and UV sources. High temperatures and open flame may produce phosgene.
Incompatibility with metals such as: aluminum powders, potassium, sodium, and zinc powder. Avoid unintended contact with amines. Avoid contact with strong bases and strong oxidizers. Avoid prolonged contact with or storage in aluminum or its alloys.
OSHA action level: 12.5 ppm. PEL is 25 ppm. (If you can smell it, the amount is too high.)

8.2 Cyclohexane - B & J Pesticide quality or equivalent.

DANGER:

All manipulations involving cyclohexane must be performed within a fume hood. Cyclohexane is highly flammable and may cause flash fire or explode. It has a flash point of -22 °C and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity.) Cyclohexane can react vigorously with oxidizing materials.
Avoid breathing vapors. Exposure can cause coughing, dizziness, headache, nausea, unconsciousness, and shortness of breath, numbness of extremities, and intoxication. Cyclohexane affects the central nervous system, having a narcotic effect.
Avoid skin contact. Cyclohexane is absorbed through the skin and is an irritation to skin and eyes. Prolonged or repeated skin contact can cause irritation and dermatitis, through defatting of skin.

8.3 GPC Calibration Solution: Solution in methylene chloride

Compound	**mg/L**
Corn Oil	25,000
bis(2-ethylhexyl) phthalate	1,000
Methoxychlor	200
Perylene	20
Sulfur	80

CAUTION: Sulfur is not very soluble in methylene chloride; however, it is soluble in warm corn oil. Weigh out the corn oil, warm it and transfer the weighed amount of sulfur into the warm corn oil. Mix it and then transfer the solution into a volumetric flask with methylene chloride, along with the other calibration compounds. Store solution at 4°C and protect from light. Make sure calibration solution comes to room temperature and is thoroughly mixed before loading onto GPC, since some analytes may precipitate when refrigerated.

WARNING: Many of the compounds used to make up mixed standards are extremely toxic. The practicality of providing hazard evaluation precludes supplying hazard warnings on each substance. Also, the interactive affect of the compounds make providing an overall evaluation impossible. However, most are at very low concentrations. The standards are also handled in a manner such that the likelihood of contact is very small. **The greatest hazard is probably associated with the ability of the solvent used to carry the material through the skin.** There should be little health risks if handled in accordance with safe working practices.

8.4 Pesticide Calibration Solution: Solution in methylene chloride

Analyte	**µg/mL**	**Analyte**	**µg/mL**

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alpha BHC	0.50	Endosulfan I	0.50
Beta BHC	0.50	Endosulfan II	0.50
Delta BHC	0.50	Endrin Ketone	0.50
Gamma BHC	0.50	Endrin Aldehyde	0.50
Aldrin	0.50	4,4' DDE	0.50
Heptachlor	0.50	4,4' DDD	0.50
Heptachlor Epoxide	0.50	4,4' DDT	0.50
Endrin	0.50	Methoxychlor	0.50
Dieldrin	0.50		

WARNING: See warning under previous standard, part 8.3.

8.5 PCB Calibration Solution: Solution in methylene chloride

Analyte	**µg/mL**	**Analyte**	**µg/mL**
Aroclor 1260	0.10	Aroclor 1016	0.10

WARNING: See warning under standard under part 8.3.

9.0 PROCEDURE

9.1 It is important not to have drastic changes in laboratory temperature (± 5°C) during an entire GPC run. If temperatures are not consistent, retention times will shift and the dump and collect times determined by the calibration standard will no longer be appropriate. Do not use equipment near the GPC that may alter the temperature of the surroundings.

9.2 COLUMN PREPARATION

9.2.1 Weigh 70.0 ± 0.1 g of Bio Beads S-X3 in a 400 mL beaker. Transfer beads to a 500-mL separatory funnel and add 300 ± 10 mL 25:75 (v/v) methylene chloride:cyclohexane. Swirl the container to ensure the wetting of all beads. Allow the beads to swell a minimum of 2 hours, maintaining enough solvent to sufficiently cover the beads at all times.

9.2.2 Turn the column upside down from its normal position and remove the inlet bed support plunger (the inlet plunger is longer than the outlet plunger.) Position and tighten the outlet bed support plunger as near the end as possible, making sure the plunger is pushed in far enough to make a good seal when tightening; approximately 2.5 - 5 cm (measured from the gel packing to the collar).

9.2.3 Raise the end of the outlet tube to keep the solvent in the GPC column. Place a small amount of solvent in the column to minimize the formation of air bubbles at the base of poured column packing.

9.2.4 Swirl the bead/solvent slurry to get a homogeneous mixture. Drain the excess solvent directly into the waste beaker and then start draining the slurry into the column by placing the separatory funnel tip against the column wall. This will help to minimize bubble formation. Swirl occasionally to keep the slurry homogeneous. Drain enough to fill the column. Place the tubing from the column outlet into a waste beaker below the column, and allow the excess solvent to drain. Raise the tube to stop the flow and close the stopcock when the top of the gel begins to look dry. Add additional solvent to just re-wet the gel.

9.2.5 Wipe any remaining beads and solvent from the inner walls of the top of the column with a laboratory tissue. Insert the plunger and make the seal just tight enough so that any beads on

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the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward. Do not tighten the column/plunger seal if beads get pushed between it and the glass column as this could damage the Teflon seal and cause leaking.

CAUTION: Do not tighten the seal if beads are between the seal and the glass surface because this may damage the seal and cause leakage.

9.2.6 Compress the column as much as possible without applying excessive force. Loosen the seal and gradually pull the inlet bed support plunger back out and rinse it and the sides of the glass column barrel with minimal solvent. Rinse and wipe any remaining beads and solvent from the plunger. Slurry any remaining beads and transfer them into the column. Repeat 9.2.5 and insert plunger. If the plunger cannot be inserted and pushed in without allowing beads to escape around the seal, continue compression of the beads without tightening the seal; remove the plunger and rinse as previously. Repeat this procedure until the plunger is successfully inserted. **Do not tighten the column/plunger seal if beads get pushed between it and the glass column.**

9.2.7 Push the plunger until it meets the gel, then compress the column bed 1 - 2 cm.

9.2.8 Connect the column inlet to the solvent reservoir (the reservoir should be placed higher than the top of the column) and place the column outlet tube in a waste container. Placing a restrictor in the outlet tube will force air out of the column more quickly. A restrictor can be made from a piece of capillary stainless steel tubing of 1.6 mm (1/16") OD x 0.025-mm (0.001") ID x 5.1 cm (2"). Pump methylene chloride through the column at a rate of 5 - 6 mL/min. for 1 to 2 hours.

9.2.9 After washing the column for 1 to 2 hours, connect the column outlet tube, without the restrictor, to the inlet side of the UV detector. Connect the system outlet to the outlet side of the UV detector. A restrictor in the outlet tube from the UV detector will prevent bubble formation that causes a noisy UV baseline. The restrictor will not affect flow rate. After pumping methylene chloride through the column for an additional 1-2 hours, adjust the inlet bed support plunger until approximately 35 - 70-kPa (5-10 psi) backpressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

9.2.10 When the GPC column is not in use, change the valve from inline to bypass. (This connects the column outlet line to the column inlet, preventing column drying and/or channeling.) If channeling occurs, the gel must be removed from the column, re-swelled and re-poured as described above. If drying occurs, methylene chloride should be pumped through the column until the observed column pressure is constant and the column appears wet. Always re-calibrate after column drying has occurred to verify retention times have not changed.

9.3 CALIBRATION OF THE GPC COLUMN FOR RESOLUTION

9.3.1 Connect the 254-nm ultraviolet detector and the Soltec Z10 integrator to the GPC.

9.3.2 Press the INDEX key on the GPC keypad to begin the pump. If the GPC has not been run in the previous 2 days, let the pump run for 2 – 3 hours. Verify the flow rate by collecting column eluate for 3 minutes ± 3 seconds in a 25-mL graduated cylinder and measure the volume, which should be 14-18 mL (4.6-6.0 mL/min.). Adjust the column pressure until the flow rate is in this range.

NOTE: When adjusting the column pressure, wait at least 5 minutes before measuring flow rate to allow the system time to equilibrate.

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9.3.3 Load the 10 ± 0.2 mL calibration solution into sample tube # 2.

9.3.4 Index through the commands and select # 5 when asked to enter the program number.

9.3.5 Starting and terminal sample should be 02.

9.3.6 Integrator parameters:

a. Check the following parameters before starting integrator

PARAMETER	SETTING	UNITS
Variab/Calib	Variab	
mV/V	mV	
Voltage	5	mV
Chart speed	30	cm/hr

b. Push chart toggle to "forward" (▼) on the integrator when calibration solution injects.

NOTE: After sample loop fills and vessel empties, valve will rotate to place sample on column, signifying injection. LED will indicate "Dump Time 1:30" and begin counting down.

9.3.7 Resolution Criteria:

a. Peaks should be symmetrical for all compounds in the calibration solution.

b. Corn oil and phthalate peaks must exhibit > 85 % resolution (where resolution is the difference between the height of the corn oil peak from the baseline and the height of the valley from the baseline divided by the height of the corn oil peak).

c. Phthalate and methoxychlor peaks must exhibit > 85 % resolution.

d. Methoxychlor and perylene peaks must exhibit > 85 % resolution.

e. Perylene and sulfur peaks must not be saturated and must exhibit > 90 % baseline resolution.

9.4 ESTABLISHING COLLECT/DUMP TIME FOR SEMIVOLATILE.

Using the information from the UV trace, establish appropriate collect and dump-time periods to ensure collection of all target analytes. Calculate the column eluant DUMP time as the time from injection to the time just before elution of bis(2-ethylhexyl) phthalate and after the elution of the corn. The COLLECT time is the time from the end of the Dump cycle to shortly after the elution of perylene, but before sulfur elutes. Use a Wash time of 10 – 12 minutes after the elution of sulfur.

9.5 ESTABLISHING COLLECT/DUMP TIME FOR ORGANOCHLORINE PESTICIDE/PCBS.

Using the information from the UV trace, establish appropriate collect and dump time periods to ensure collection of all target analytes. Calculate a DUMP time from injection to a time which removes > 85 % of bis(2-ethylhexyl) phthalate but collects > 95 % of the methoxychlor. The COLLECT time is the time from the end of the Dump cycle to shortly after the elution of

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perylene, but before sulfur elutes. Use a Wash time of 10 – 12 minutes after the elution of sulfur.

9.6 ORGANOCHLORINE PESTICIDE/PCB CALIBRATION:

9.6.1 Load 10 ± 0.2 mL of methylene chloride into sample tube # 1 and # 2. Load 2 ± 0.1 mL of pesticide calibration solution with 8 ± 0.1 mL methylene chloride into sample tube # 3. Load 1 ± 0.1 mL of PCB calibration solution with 9 ± 0.1 mL of methylene chloride into sample tube # 4.

9.6.2 After running GPC on these four tubes, concentrate the eluant and solvent exchange to hexane, where the final volume for sample # 2 (GPC blank) is 1 ± 0.1 mL, # 2 (Pesticide check) is 5 ± 0.1 mL and #3 (PCB check) is 2 ± 0.1 mL.

9.6.3. Analyze by method AM E-8080. The recovery of all of the analytes must be 80 to 110 % before samples can be loaded onto GPC. The GPC blank must exhibit no analytes above the quantitation limit of the analytical method*. If the blank does not meet, pump additional methylene chloride through the GPC for 1-2 hours before analyzing another GPC blank. A blank that is run after the resolution check will always exhibit significant amounts of methoxyclor.

*** See SOP for Detection and Quantitation Limits for exceptions to this.**

9.7 EXTRACT PREPARATION

9.7.1. Pre-rinse calibrated c-tubes, glass syringes and sample tubes. Rinse the barrels with the valve in place. Rinse the plungers separately.

9.7.2 Reduce the sample extract volume to 6 – 8 mL and transfer to a 15-mL calibrated centrifuge tube. Since soils are extracted with 1+1 (v/v) methylene chloride/acetone, they should first be concentrated to 1 ± 0.5 mL. Adjust extract volume to 10 ± 0.2 mL with methylene chloride. Thoroughly mix using a vortexer before proceeding (if the extracts were removed from the refrigerator, let them warm to room temperature, adjust volume if necessary, then mix).

9.7.3 If a noticeable amount of sediment exists, it may be helpful to centrifuge extract to prevent excessive plugging of filters. Weigh capped c-tube containing extract. Create a counterbalance capped c-tube with the appropriate amount of methylene chloride to match the mass of the extract c-tube within 0.5 g. Place the c-tubes directly opposite each other into the Clay Adams Safety-Head centrifuge and set speed to 3 – 4. Centrifuge sample for 3 – 5 minutes, then turn setting to zero. Allow centrifuge to stop and remove extract.

9.7.4 Place the valve on the syringe, followed by a 0.45-um PTFE filter disk. If the extract appears to have suspended solids or does not centrifuge well, a 1.0-um filter disk may be placed on the syringe followed by the 0.45-um disk. The 1.0-um disk does not plug as easily and acts as a pre-filter for the 0.45-um disk. Transfer the entire extract to the syringe, minus sediment, making sure that the valve is closed and held in place over the GPC sample tube. The sample tube should be held against the filter disk with light pressure to keep the valve from leaking or dislodging from the syringe. Open the valve and slowly discharge the extract into the sample tube. If there is an increase in the plunger resistance, the filter may be getting plugged. If this is the case, carefully remove the plunger, re-insert into the barrel, and gently apply pressure. This may be repeated several times to try and establish flow again. In some instances, it may be necessary to close the valve, replace the filter, and continue the transfer until the syringe is fully discharged. (In the case of where there is limited sample, 8.0 mL is the minimum amount of sample that can be loaded into the GPC sample tubes.)

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9.7.5 On tissue samples or oil waste, loading onto the GPC is a concern. The column has a maximum capacity of 0.5 g of oil/lipids.

a. In the case of tissue samples, a lipid determination is done before GPC clean up. From the 10.0 mL volume, take 1±0.1 mL and place in a pre-weighed, to the nearest 0.0001g, aluminum pan. Place in the hood and allow the methylene chloride to evaporate. Weigh the pan + residue and calculate and record on a data sheet the amount of residue. Multiply the amount of residue by 10. If the result is greater than 0.5 g, then the extract needs to be diluted before loading onto the GPC.

b. In the case of waste samples containing high amounts of oil, load only 0.5 grams of sample onto GPC.

9.7.6 Place the sample tube on the GPC. Make sure that the tube is snug in the holder. If there is an air leak in the fitting of the tube to the GPC, the sample will not draw up successfully into the GPC and the GPC will halt on that sample.

a. Load samples onto the GPC from clear samples to dark samples. While color may not indicate the content of the sample, it is good practice the run the cleaner-looking samples first.

b. If the sample is highly colored (or is suspected of having a high concentration of analytes of interest), methylene chloride blanks should be loaded after these samples to minimize carryover.

c. A GPC blank is run prior to initiation of clean up (Sample port # 1). This acts as a system flush.

9.7.7 Place the 250-mL Erlenmeyer flask on the corresponding collection outlet (indicated by the numbered tag on the outlet tube). Place the sample label on this Erlenmeyer flask and record the slot number and sample number in the GPC log.

9.7.8 Make sure that the methylene chloride and the line rinse reservoir are full. Also, check that the waste bottle is empty.

9.8 GPC CLEAN UP

9.8.1 Column-bypass valve should be in the in-line mode. Turn on the rinse pressure located near the back of the instrument; it should read 70 kPa (10 psi.) Turn on power to the GPC control panel. System Preparation Display Messages will read:

AS2000 -- ABC LABORATORIES -- (C) 1991
SOFTWARE VERSION=6016-PC, 09-05-91/LLS
SYSTEM TEST IN PROGRESS . . . PLEASE WAIT
ALL LEDS SHOULD BE LIT . . . PLEASE CHECK
TESTING COMPLETE, SYSTEM OPERATIONAL
If self test fails, an error message will be displayed. Refer to troubleshooting section of manual for information about the error message.

9.8.2 SELECT HIGHER PRESSURE LIMIT (NO=0) -0?

Accept the lower limit default by pressing the INDEX key.

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9.8.3 FILL INPUT MODULE RESERVOIRS

This is to remind you to fill the tanks when necessary. The solvent pump will turn on at this time. If the tanks have not been filled, fill them before proceeding. Press the INDEX key.

9.8.4 PERFORM A RINSE LOOP PURGE

The rinse loop should be purged whenever the rinse solvent is first added to an empty rinse reservoir or if it is necessary to insure that the rinse loop is full of rinse solvent prior to sample processing. Press the INDEX key.

9.8.5 ULTRASONIC SNSR PRE-LOAD TIME = :05?

If the sensor does not detect start of sample before PRE-LOAD TIME has expired, an error message results. Press the INDEX key to accept the default of 5 seconds.

9.8.6 ULTRASONIC SNSR DEBOUNCE TIME = :00.05?

Allows for the passage of minute air bubbles in the sample without triggering a false end-of-sample detection. If liquid is not detected before the DEBOUNCE TIME expires, an error condition is generated and sample processing halts. Press the INDEX key to default at 0.05 seconds.

9.8.7 PUT COLLECT LINES IN SAMPLE VESSELS

A clean receiving vessel (such as a 125-mL Erlenmeyer flask) is needed at the output tube for each input position that will have a sample to be processed. Press the INDEX key.

9.8.8 IS CALIBRATE MODE DESIRED?

Calibrate mode is used when analyzing fractions of a sample. See the operation manual for more information.

Press the INDEX key.

9.8.9 LOAD SAMPLE INPUT POSITIONS

Transfer sample into a 10.0-mL glass syringe that has a mini-nert valve and 0.45-µm Acrodisc filter attached. At least 7 mL of filtered sample is needed for proper operation, but 10.0 mL is the preferred volume. Filter sample into the 15-mL GPC tube and screw onto the proper sample container support arms. Tighten each tube until the sample starts to rise in the pick-up tube.

CAUTION: Mount the sample containers uniformly finger tight. Do not over tighten because of possible damage that occurs to sample tubes and loss of pressure during sample loading.
The GPC Blank is always in the # 1 sample position and samples are loaded according to color (light to dark). A second GPC blank may be put on at the end of a sample run to determine if there is any contamination to the column.

Sample position number 2, 11, 15, and 23 take much longer to load than the other positions. It is recommended that these positions be loaded with methylene chloride only during the sample GPC run.

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9.8.10 MODE = GPC ONLY, CURRENT PROGRAM = #*?

Program # 1 = BNA
Program # 2 = Pesticides
Program # 3 = Rinse Cycle
Program # 4 = Not Currently Utilized
Program # 5 = GPC Calibration

This will default to the last program saved; therefore, accept the program by pressing the INDEX key, or select another program by pressing the corresponding number and pressing ENTER and then INDEX.

9.8.11 ULTR-SONIC SNSR MAX LOAD TIME = 2:00.0?

If the ultrasonic sensor does not detect end of sample before this time has elapsed, processing stops and an error message is issued. Press the INDEX key.

9.8.12 DUMP TIME = **:**?

The last dump time stored for the current program is displayed **:**?. Enter desired time if different from current program followed by pressing the ENTER and INDEX keys.

9.8.13 COLLECT TIME = **:**?

The last collect time stored for the current program is displayed **:**? Enter desired time if different from current program followed by pressing the ENTER and INDEX keys.

9.8.14 TOTAL WASH TIME = **:**?

The last wash time stored for the current program is displayed **:**?. Enter desired time if different from current program followed by pressing the ENTER and INDEX keys.

9.8.15 START WITH SAMPLE # = **?

The last starting sample number used is displayed **?. Enter the desired starting sample number, followed by pressing the ENTER and INDEX keys.

9.8.16 TERMINAL SAMPLE # = **?

The last terminal sample number used is displayed **?. Enter the desired terminal sample number, followed by pressing the ENTER and INDEX keys. The terminal sample number may be changed during sample processing.

9.8.17 PROCESS WITH MULT PROGRAMS (NO=0) -0?

Up to five different programs can be used during sample processing. See the operation manual for more information.

Press INDEX key.

9.8.18 CHECK THE SOLVENT FLOW RATE

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Check that the flow rate measure 5.0 – 6.0 mL/min and that the column pressure is within operating parameters (5 – 10 psi). Press the INDEX key.

9.8.19 EMPTY WASTE CONTAINERS BEFORE STARTING

Reminder to empty the waste container if it has not been done already. Press INDEX key.

9.8.20 SMPLS = **:** MODE = GPC ONLY PUSH RUN?

Sample numbers have been previously programmed in during this process. Double check to see if column pressure is approximately 35-70 kPa (5-10 psi), column-bypass valve is in line, reservoirs are full of solvent, and collection vessels are under the correct output tube. It is possible to INDEX through this command to adjust any parameters, if needed. Push RUN when ready.

9.8.21 DOCUMENT SAMPLE NUMBERS AND VOLUMES IN LOG BOOK

9.9 COLLECTION OF SAMPLE AFTER CLEANUP

After collection before unloading, double check to see that sample numbers that were loaded correspond to collection vessel numbers.

9.10 Remember that only half of the sample extract is processed by the GPC (5 mL of the 10-mL extract is loaded onto the GPC column, whereas 3 mL minimum is used to flush the lines), and thus a dilution factor of 2 must be used for quantitation of the sample in the determinative method.

9.11 To minimize contamination, it is a good idea to run solvent blanks in all the just completed sample vessels after collection, or just prior to the next GPC cleanup.

9.11.1 Load 10 ± 0.5 mL of methylene chloride into pre-rinsed sample tubes and place on GPC. Check reservoirs to make sure ample solvent is available and empty waste container. Place collection lines in a plastic beaker to contain waste solvent.

9.11.2 Choose program # 3 – rinse cycle. Verify the following program times.

ULTR-SONIC SNSR MAX LOAD TIME = 2:00.0
DUMP TIME = 2:00
COLLECT TIME = 1:00
TOTAL WASH TIME = 1:00

9.11.3 Enter the start and terminal sample numbers. Press RUN when prompted.

9.11.4 Upon completion of the rinse program, switch column bypass valve to the “bypass” position. Properly empty or dispose of waste containers.

10.0 QUALITY ASSURANCE

10.1 The analyst must demonstrate that the compounds of interest are being quantitatively recovered by the clean-up technique before the clean-up is applied to samples. The resolution mix and solvent blank must be run within the past 7 days before any samples are processed. The calibration criteria in part 9.3.7 must be met before samples are loaded and run. The pesticide and PCB calibration mix must also be run within the past 7 days and must meet the calibration

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criteria in part 9.6 before any organochlorine pesticide or PCB samples are loaded and analyzed.

10.2 As in all methods, the associated quality control samples (Blanks, LCS, and matrix spikes) must also be processed through this clean-up procedure.

10.3 METHOD PERFORMANCE

Refer to SW 846 noted in the reference (Part 13) for GPC elution and recovery data.

11.0 REPORT

Data is not generated by this procedure so there is no report. Data necessary for documentation, such as the volume in 9.7.4.a. and the color in 9.7.5, must be documented either in a logbook or with the data from the determinative methods.

12.0 KEY WORDS

GPC, Gel-permeation, cleanup, method 3640

13.0 REFERENCES

"Test Methods for Evaluating Solid Waste SW846", Volume 1B: Laboratory Manual Physical/Chemical Methods, Revision 2, January 1995, page 3640A -16 to 20.

Or visit http://www.epa.gov/epaoswer/hazwaste/test/pdfs/3640a.pdf

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W-19

Condensation and Evaporation Techniques

Process Owner (TS/PM/OM/LM)	
Randy Eatherton	
Assignment	Reviewed June 23, 2003 by Christine Devine Reviewed August 19, 2008 by Christine Devine
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1.0 SCOPE

1.1 Following are two procedures to concentrate organic solvents after extraction to prepare for analysis. Rate of evaporation will be influenced by the boiling points of the solvents and the levels of contamination of the samples.

2.0 SUMMARY OF PROCEDURE

2.1 Evaporation using a Kuderna-Danish (K-D) apparatus:

A sample extract is heated in a flask with an attached condenser over a steam bath in order to evaporate excess solvent while retaining analyte compounds.

Solvent exchange may be performed during this condensation.

2.2 Evaporation using nitrogen gas:

Extracts may be further concentrated using a gentle stream of N_2 gas to evaporate excess solvent while retaining analyte compounds.

Solvent exchange may be performed during this concentration.

3.0 INTERFERENCES

High boiling points of solvents may interfere with efficient evaporation on the steam bath.

Higher density of undesired solvent will cause difficulty in evaporating when doing solvent exchange.

4.0 APPARATUS AND MATERIALS

4.1 Kuderna-Danish (K-D) apparatus.

4.1.1 Concentrator tube -- 15-mL, graduated, with Air Jacket (Kontes).

4.1.2 Evaporation flask -- 500-mL (Kontes K-570001-500) or as specified in method. Attach to concentrator tube with clamps.

4.1.3 Snyder column -- Three ball macro (Kontes K-503000-0121).

4.2 Steam bath -- With concentric ring cover. Use the bath in a hood.

4.3 The Meyer N-Evap Organomation Analytical Evaporator, Model No. 112.

4.4 Plastic retainer clips to hold apparatus together.

4.5 Teflon Boiling chips.

5.0 REAGENTS

Refer to the specific test method for reagents needed.

6.0 PROCEDURE

6.1 Concentration of extract using Kuderna-Danish Apparatus

6.1.1 Assemble Kuderna-Danish (K-D) flask and collector (see appropriate test method for flask size), using the K-D clip to secure the joint. Rinse with appropriate solvent three times and place upside down in steam-bath clamp in the hood to dry.

6.1.2 Secure the flask with the clamp on steam bath and slowly quantitatively transfer sample extract into K-D flask, rinsing the sample container three times with the appropriate solvent.

NOTE: If Na_2SO_4 was used to dry the extract, a funnel packed with glass wool can be used in the transfer to keep Na_2SO_4 out of the extract. Make sure to rinse the glass wool and funnel with solvent into the KD flask to complete the quantitative transfer.

6.1.3 Add one or two pre-cleaned boiling chips to the flask.

6.1.4 Pre-wet a three-ball Snyder column by adding about 1 mL of appropriate solvent to the inside of the inverted column. Rinse the ground-glass joint and attach the Snyder column to the top of the K-D apparatus.

6.1.5 Place the lower portion of the receiving tube of the K-D apparatus into the steam bath. Adjust the steam heating so that the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus (to insure even drainage of the condensor into the KD flask). Adjust the steam flow so that at a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. If the final solvent will be hexane (such as AM E-608, 8081 or 8082), then add 1 mL of hexane into the top of the condensor. This will act as a keeper for low boilers.

NOTE: If there is not enough heat generated with the steam valve open all the way (i.e. when hexane is being evaporated), wrap aluminum foil around the base of the apparatus to keep all the hot vapor concentrated at the base of the K-D flask.

6.1.6 When the top of the condensor is dry (i.e., no wetting of the top of the condensor with vapors), remove the K-D apparatus from the steam bath and allow it to drain and cool for at least 10 min. and no more than 4 hr. If the solvent draining from the condensor is not evenly wetting the KD, adjust the vertical position of the apparatus. After cooling, the final volume will be about 4 mL.

6.1.7 Remove the Snyder column. While holding the collector, remove K-D clip and wipe area with a kimwipe or paper towel, making sure to absorb all water at joint area. Remove collector tube, using the appropriate solvent to rinse the ground glass joint of the flask into the collector, and put the collector in the test tube rack. If storing extracts for more than overnight, stopper the collectors with Teflon stoppers or transfer the samples to 15-mL centrifuge tubes and cap. Aluminum foil can be

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used to cover the collectors, but only if the sample is going to be transferred in less than 24 hours. Store appropriately.

6.2 NITROGEN EVAPORATION

6.2.1 Transfer the sample to 15-mL centrifuge tube before nitrogen evaporation using a Pasteur pipette with three rinses of the collector tube with appropriate solvent.

6.2.2 Wash needles on nitrogen evaporator (N-evap) with solvent and wipe down with kimwipe. If heat is desired, turn on the water bath and adjust to the appropriate temperature. Avoid using heat if possible. The cooling effect of evaporation helps retain low boilers.

6.2.3 Place samples on N-evap and carefully secure with spring. Be sure N-evap flow meter is off. Lower needles to one to two inches above liquid level. Open all needle valves to approximately the same level.

6.2.4 Turn nitrogen on at the tank. Adjust the flow at the flow meter to a rate sufficient to make a small dimple in the surface of the liquid and not splash the sample (there should be no audible splashing sound). Check all samples for proper evaporation rate.

NOTE: When needle valves are opened or closed during evaporation, the flow rate to all needles will be affected.

6.2.5 Do not allow the samples to go dry unless it is specified in the appropriate method. Reduce the sample to ½ the volume specified in the method. During transfer, the remaining volume will be used to rinse the centrifuge tube to bring to final volume.

6.2.6 If the solvent is to be exchanged, reduce the volume to ¼ of the specified final volume. Add the final solvent and reduce to ¼ of the specified volume and repeat this process. In the case of exchanging methylene chloride to hexane, the hexane floats above methylene chloride and requires periodic mixing.

7.0 KEY WORDS

N-evaporator, KD apparatus, condensation, evaporation

8.0 REFERENCES

EPA manual SW-846

9.0 REVISION HISTORY

4/10/04 – Changed SOP code from AP G-CONC**.**

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W-20

Organic Laboratory Analytical Standards

Process Owner (TS/PM/OM/LM)	
Randy Eatherton	
Assignment	Reviewed July 18, 2007 by Christine Devine
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1.0 SCOPE

This standard operating procedure covers the guidelines and protocols concerning standards preparation within the organics lab.

2.0 SUMMARY OF METHOD (or SOP)

Standards prepared and/or used in the laboratory will be documented in appropriate "Standard" logbooks, extraction logbooks and instrument logbooks and be traceable to their source.

3.0 INTERFERENCES

See method for which the standards are being made.

4.0 ESTIMATE OF ANALYTICAL or TESTING TIME

See method for which the standards are being made.

5.0 AMOUNT OF SAMPLE REQUIRED

Not applicable.

6.0 SAMPLING, SAMPLE HANDLING, AND PRESERVATION

Not applicable.

7.0 EQUIPMENT REQUIRED

See method for which the standards are being made.

8.0 REAGENTS REQUIRED

See method for which the standards are being made.

9.0 PROCEDURE

9.1 Standard Identification

9.1.1 The date a neat chemical or standard mixture is received, and an expiration date will be written on the container upon receipt. If the expiration date is not known, a general guideline of one year from date of receipt may be used. Any standards prepared from these chemicals will retain the expiration date. (The method for which the standard is prepared will state the length of usability unless the stability of the mix is an issue.)

9.1.2 When an analytical standard is prepared, the following information will be entered into an appropriate standard log:

a. Laboratory designated standard number.
b. Standard name.
c. Standard lot number if known.
d. Standard purity if known.
e. Date prepared and/or date initially used.
f. Preparers initials.
g. Solvent the standard is made in
h. Calculations showing how the final concentration was derived.
i. Expiration date

9.1.3 The standard number for any standard used for instrumental analysis (e.g. internal standards, calibration standards, matrix spiking standards etc.) will be entered into the instrument run log to provide traceability to the source of the standard.

9.2 Numbering Convention

9.2.1 Standards used for volatiles analyses will be recorded into logbooks that will be stored in SLM233. References to standards in the logs will include the standard log volume number, the page number and the standard number.

For example, V2-10-5, where

V2 - indicates volatiles standards volume 2
10 - indicates page 10 of the specified volume
5 - indicates the 5th standard listed on page 10

9.2.2 Standards for all GC and GC/MS analyses will be recorded in logbooks that will be stored in the east alcove in 211 SLM. References to standards in the logs will be designated using sequential numbers, month and year.

For example, 708005, where

the first digit indicates the last digit of the year (2007 in this example)
the second and third digit the month (August)
the last three digits are sequential for the standards made in that month

9.3 Standard Storage

9.3.1 Neat chemical standards known or suspected of being unstable will be kept in refrigerated storage.

9.3.2 Standard solutions should be stored at 4ºC in sealed ampules or solvent rinsed amber glass containers with PTFE lined caps. The headspace above the solution should be minimized. Purchased standard solutions may be stored at room temperature if stated by the manufacturer.

9.4 Priority Pollutant Standards Preparation Observations

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9.4.1 Volatiles: The "gases" in volatiles standard mixes are susceptible to loss; hence these standards should be monitored carefully.

9.4.2 Semivolatiles: Semivolatiles standards are purchased from vendors as a series of mixes. The mixes should not be combined until immediately before sample analysis. Mixed standards should not be used for more than two weeks following preparation.

9.4.3 Pesticides: Pesticides and Aroclors are very stable, and are made in iso-octane and stored at room temperature.

9.5 Standard Verification

9.5.1 Whenever a new standard is made, a cross verification with the previous standard is done before placing the new standard into production.

9.5.2 Verification of the standard curve is done with each batch with a lab control spike (the LCS goes through the sample preparation) or/and an Initial Calibration Verification (CCV). Both of these standard checks are from a 2nd source vendor (or in the case where there is only one vendor, a different lot).

10.0 QUALITY ASSURANCE

See method for which the standards are being made.

11.0 REPORT

Not applicable.

12.0 KEY WORDS

neat standards, chemical mixture, storage, logbook, pesticides

13.0 REFERENCES

See method for which the standards are being made.

14.0 REVISION HISTORY

14.1 7/18/07 – Method updated to new format and updated logbook information.

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W-21

VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

Process Owner (TS/PM/OM/LM) **Tom Wasnock**	
Assignment	Reviewed on February 10, 2004 by Christine Devine Reviewed on August 27, 2008 by Christine Devine
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1.0 SCOPE

1.1 Method 8260 is used to determine volatile organic compounds, primarily in ground and surface water. This method refers only to applications involving aqueous samples, however, this method can be applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds are determined by our application of this method:

Compound	CAS No.	Method 5030
Acetone	67-64-1	pp
Benzene	71-43-2	c
Bromobenzene	108-86-1	na
Bromochloromethane	74-97-5	c
Bromodichloromethane	75-27-4	c
4-Bromofluorobenzene(surr)	460-00-4	c
Bromoform	75-25-2	c
Bromomethane	74-83-9	c
2-Butanone(MEK)	78-93-3	pp
n-Butylbenzene	104-51-8	na
sec-Butylbenzene	135-98-8	na
tert-Butylbenzene	98-06-6	na
Carbon disulfide	75-15-0	pp
Carbon tetrachloride	56-23-5	c
Chlorobenzene	108-90-7	c
Chlorobenzene-d_5 (surr)	na	c
Chlorodibromomethane	124-48-1	c
Chloroethane	75-00-3	c
Chloroform	67-66-3	c
Chloromethane	74-87-3	c
2-Chlorotoluene	95-49-8	na
4-Chlorotoluene	106-43-4	na
1,2-Dibromo-3-chloropropane	96-12-8	pp
1,2-Dibromoethane	106-93-4	c
Dibromomethane	74-95-3	c
1,2-Dichlorobenzene	95-50-1	c
1,3-Dichlorobenzene	541-73-1	c
1,4-Dichlorobenzene	106-46-7	c
1,4-Dichlorobenzene-d_4 (IS)	na	c
Dichlorodifluoromethane	75-71-8	c

Compound	CAS No.	Method 5030
1,1-Dichloroethane	75-34-3	c
1,2-Dichloroethane	107-06-2	c
1,2-Dichloroethane-d_4(surr)	na	c
1,1-Dichloroethene	75-35-4	c
cis-1,2-Dichloroethene	156-59-2	na
trans-1,2-Dichloroethene	156-60-5	c
1,2-Dichloropropane	78-87-5	c
1,3-Dichloropropane	142-28-9	na
2,2-Dichloropropane	594-20-7	na
1,1-Dichloropropene	563-58-6	na
cis-1,3-Dichloropropene	10061-01-5	c
trans-1,3-Dichloropropene	10061-02-6	c
Ethylbenzene	100-41-4	c
Fluorobenzene(IS)	462-06-6	c
Hexachlorobutadiene	87-68-3	c
2-Hexanone	591-78-6	pp
Isopropylbenzene	98-82-8	c
p-Isopropyltoluene	99-87-6	na
Methyl-t-butyl ether	1634-04-4	na
Methylene chloride	75-09-2	c
4-Methyl-2-pentanone(MIBK)	108-10-1	pp
Naphthalene	91-20-3	c
n-Propylbenzene	103-65-1	na
Styrene	100-42-5	c
1,1,1,2-Tetrachloroethane	630-20-6	c
1,1,2,2-Tetrachloroethane	79-34-5	c
Tetrachloroethene	127-18-4	c
Toluene	108-88-3	c
Toluene-d_8 (surr)	2037-26-5	c
1,2,3-Trichlorobenzene	87-61-6	na
1,2,4-Trichlorobenzene	120-82-1	c
1,1,1-Trichloroethane	71-55-6	c
1,1,2-Trichloroethane	79-00-5	c
Trichloroethene	79-01-6	c
Trichlorofluoromethane	75-69-4	c
1,2,3-Trichloropropane	96-18-4	c
1,2,4-Trimethylbenzene	95-63-6	na
1,3,5-Trimethylbenzene	108-67-8	na
Vinyl chloride	75-01-4	c
o-Xylene	95-47-6	c
m-Xylene	108-38-3	c
p-Xylene	106-42-3	c

c = Adequate response by this technique
na = Not available
pp = Poor purging efficiency resulting in high Estimated Quantitation Limits
surr = Surrogate
IS = Internal Standard.

1.2 There are various techniques by which these compounds may be introduced into the GC/MS system. The techniques used by the laboratory are purge-and-trap, by Method 5030 (aqueous

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samples) for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. Method 5000 provides more general information on the selection of the appropriate introduction method.

1.3 EPA Method 8260B can be used to quantitate most volatile organic compounds that have boiling points below 200 ºC. Volatile, water soluble compounds can be included in this analytical technique; however, detection limits may be higher due to poor purging efficiency. The following compound types may also be amenable to analysis by Method 8260: low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. See Table 1 for analytes and retention times that have been evaluated on a purge-and-trap GC/MS system. Also, the method detection limits for 25-mL sample volumes are presented.

1.4 The estimated quantitation limit (EQL) of Method 8260 for an individual compound is somewhat instrument dependent and also dependent on the choice of sample preparation/introduction method.

Using standard quadrapole instrumentation and the purge-and-trap technique, limits are approximately 0.5 µg/L for ground water. EQLs will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.

2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method and the analytes are introduced directly into a wide-bore capillary column before being flash evaporated to a narrow-bore capillary column for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC).

2.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection interfaced to the ion source. Identification of target analytes is accomplished by comparing their mass spectra with the electron-impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a calibration curve containing a minimum of five points.

2.3 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components are avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation

3.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

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For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105 °C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning.

Many analytes exhibit low purging efficiencies from a 25-mL sample. This often results in significant amounts of these analytes remaining in the sample purge vessel after analysis. After removal of the sample aliquot that was purged, and rinsing the purge vessel three times with organic-free water, the empty vessel can be subjected to a heated purge cycle prior to the analysis of another sample in the same purge vessel. This will reduce sample-to-sample carryover.

3.5 Special precautions are taken to analyze for methylene chloride and other common laboratory solvents. Purge and trap methanol is the only solvent stored in the laboratory and the volatile sample storage area is isolated from all sources of solvents. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing are constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination. There is the potential that the lab air could become contaminated with solvent from nearby laboratories or through the building ventilation system.

3.6 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

3.7 The use of sensitive mass spectrometers to achieve lower detection levels increases the potential of detecting laboratory contaminants as interferences.

4.0 ESTIMATE OF ANALYTICAL or TESTING TIME

Single sample analysis time is approximately 30-40 min. Sample preparation time can vary between 5 and 30 min depending on sample condition. Low-level water samples need only be measured to 25 mL and spiked with surrogate and internal standards.

Autosampler capability allows the VOA lab to analyze approximately 20 samples per analytical shift, depending on instrument run time. The time required for data reduction may vary between 5-60 min per sample depending on reporting requirements and sample complexity.

5.0 AMOUNT OF SAMPLE REQUIRED

For water samples, a minimum of 25 mL is required for analysis. However, a minimum of three 40-mL vials are suggested since additional analyses such as duplicates, re-runs, dilutions, or matrix spikes may require extra volume.

6.0 SAMPLING, SAMPLE HANDLING, AND PRESERVATION

Liquids should be sampled in standard 40-mL glass screw-cap VOA vials with Teflon-lined silicone septa. The vials should be tapped slightly as they are filled to try and eliminate as much free air space as possible. Three vials should be filled per sample location.

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When collecting the samples, liquids should be introduced into the vials gently to reduce agitation which might drive off volatile compounds. In general, liquid samples should be poured into the vial without introducing any air bubbles within the vial as it is being filled. Should bubbling occur as a result of violent pouring, the sample must be poured out and the vial refilled. The vials should be completely filled at the time of sampling, so that when the cap is fitted and sealed, and the vial inverted, no headspace is visible.

Due to differing solubility and diffusion properties of gases in liquid matrices at different temperatures, it is possible for the sample to generate some headspace during storage. This headspace will appear in the form of micro bubbles, and should not invalidate a sample for volatiles analysis.

The presence of a macro bubble in a sample vial generally indicates either improper sampling technique or a source of gas evolution within the sample. "Pea-sized" bubbles or smaller do not adversely affect volatiles data.

All samples must be preserved with the addition of 1:1 HCl to bring the sample pH to a value of 2, then iced or refrigerated at 4 °C, from the time of collection until analysis. Vials are usually shipped pre-preserved. Care must be taken not to overfill or wash out the preservative. In some cases, violent foaming will occur when samples are added to pre-preserved vials indicating dissolved carbon dioxide. Unpreserved samples should be noted on the chain of custody.

Unpreserved samples must be analyzed within 7 days of collection. The holding time is extended to 14 days if samples are acidified.

Prior to analysis, or immediately after loading a sample, check the pH using a indicating strip and record to the nearest whole number or with a range (i.e. pH <2) in the sample logbook.

7.0 EQUIPMENT REQUIRED

1. Microsyringes - 10-µL, 25-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL. These syringes should be equipped with a 20-gauge (0.015 cm or 0.006 in., ID) needle having a length sufficient to extend into the syringes used to contain samples, prior to spiking with internal standard/surrogate solution, and into the reactivials used to contain calibrator solutions.

7.2 Syringe: 25-mL.

7.3 Balances: Top-loading, capable of weighing to the nearest 0.001 g.

7.4 Volumetric flasks: 10-mL, 25-mL and 100-mL, with ground-glass stoppers.

7.5 Vial: 40-mL, or larger, equipped with a screw cap with a hole in the center.

7.6 Septum: Teflon-faced silicone

7.7 Reacti-vial: 1-mL vials with septum cap for standard solutions.

7.8 Purge-and-trap device: VOL1 is the primary system for the analysis of this method. The VOL1 GC/MS system has a Tekmar LSC 3100 equipped with a Tekmar ALS 2016 autosampling unit. . The VOL2 GC/MS system has a Tekmar LSC 3000 equipped with a Tekmar ALS 2016 autosampling unit.

7.8 Pre-packed Traps: Supelco VOCARB 3000 trap.

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7.9 Gas chromatograph: Agilent 6890 GC.

7.10 Column: JW DB-624, 30m x 0.253mm, 1.4-µm film thickness

7.11 Mass spectrometer: Agilent 5973 MSD

7.12 Data system: Currently the Chemstation software is used for data acquisition and reduction. An EPA/NIST Mass Spectral Library is currently used. The Chemserver/Target system is being evaluated for use with this method.

8.0 REAGENTS REQUIRED

8.1 Organic-free reagent water - reagent water is defined as water in which an interferant is not observed at or above the quantitation limit of the parameters of interest. Reagent water is generated by passing building de-ionized water through the following processes: Reverse osmosis system, de-ionization and carbon filtration. All references to water in this method are organic-free reagent water unless otherwise specified.

8.2 Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate. The standard logbook is used to document the preparation of all standard materials used in the VOA lab. All standard solutions are stored in the lab freezer.

Standards (mixed)

WARNING: Many of the compounds used to make up mixed standards are extremely toxic. The practicality of providing hazard evaluation precludes supplying hazard warnings on each substance. Also, the interactive effect of the compounds make providing an overall evaluation impossible. However, most are at very low concentrations. The standards are also handled in a manner such that the likelihood of contact is very small.
The greatest **hazard is probably associated with the ability of the solvent used to carry the material through the skin.**
There should be little health risks if handled in accordance with safe working practices.
Consult the MSDS sheet if there is one for the material.

8.3 Helium - UHP grade helium

8.4 Methanol, CH3OH. Baxter B&J Brand Purge and Trap Grade (stored apart from other solvents).

WARNING: All manipulations involving methanol should be performed within a fume hood. Methanol is flammable and burns with a clear, almost invisible flame. It has a flash point of 12 °C, explosive limits in air in the range of 6 - 36%, and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity). Methanol can react vigorously with strong oxidizing materials. Avoid contact with strong acids, acid chlorides, acid anhydrides, and alkali metals.
Avoid breathing vapors. Initial symptoms may be only that of mild intoxication, but may become severe after 12 to 18 hours. Methanol can be irritating to mucous membranes and upper respiratory tract. Exposure can cause headache, muscle weakness, dizziness, nausea, vomiting, narcosis, respiratory failure, low blood pressure, gastrointestinal disturbances, convulsions, and may be fatal. Methanol is a central nervous system depressant and can cause kidney, liver (enlargement), eye (especially optic nerve), and heart damage.

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Avoid skin contact. Methanol is absorbed through the skin. Prolonged or repeated skin contact can cause irritation and dermatitis.
Eye contact may cause irritation and/or temporary corneal damage.
Prolonged exposure to methanol has been known to cause fetal development abnormalities in pregnant rats.

8.5 Antifoam agent – A working solution of antifoam agent is prepared by diluting 1 ± 0.1 g of Dow Antifoam C to 100 ± 10 mL of water. The solution can be added to samples, as necessary, to inhibit foaming.

8.6 Hydrochloric acid (1+1, v/v), HCl - Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

WARNING: Handle with care. Wear safety goggles and a plastic apron and use in a hood. Conc. hydrochloric acid is a strong, corrosive acid. Liquid and vapor can cause burns. Carefully add acid to water with frequent or continuous stirring. Mix in a tub or stoppered sink. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.
Avoid breathing vapors. Exposure can cause spasm, destruction to tissue of the mucous membranes, inflammation and edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema.
Symptoms of exposure may include burning sensation, coughing, choking, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. In severe cases, inhalation may result in circulatory failure, and death.
Avoid skin contact. Hydrochloric acid can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and discolor skin.
Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Long-term exposure to concentrated vapors may cause erosion of teeth.
Highly reactive with strong bases, metals, metal oxides and alkali metals, hydroxides, amines, carbonates and other alkaline materials. Avoid contact with materials such as cyanides, sulfides, sulfites, and formaldehyde.
Mixing with strong acids may produce toxic products. Conc. sulfuric acid will dehydrate hydrochloric acid, releasing hydrogen chloride gas. Certain mixtures with nitric acid will give aqua regia, which is unstable and extremely corrosive, and gives off toxic gas as it decomposes.
OSHA PEL is 7 mg/m3 (5 ppm)

9.0 PROCEDURE

9.1 Various alternative methods are provided for sample introduction. All internal standards, surrogates, and matrix spiking compounds (when applicable) must be added to the samples before introduction into the GC/MS system. Consult the sample introduction method for the procedures by which to add such standards.

9.1.1 Purge-and-trap - This includes purge-and-trap for aqueous samples (Method 5030). Method 5035 also provides techniques for extraction of high concentration solid and oily waste samples by methanol (and other water-miscible solvents) with subsequent purge-and-trap from an aqueous matrix using Method 5030.

a. Traditionally, the purge-and-trap of aqueous samples is performed at ambient temperature, while purging of soil/solid samples is performed at 40 °C, to improve purging efficiency.

b. Aqueous and soil/solid samples may also be purged at temperatures above those being recommended as long as all calibration standards, samples, and QC samples are purged at

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the same temperature, appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Purging of aqueous samples at elevated temperatures (e.g., 40 °C) may improve the purging performance of many of the water soluble compounds which have poor purging efficiencies at ambient temperatures.

9.2 Chromatographic conditions used in our application of the method can be found in Table 2.

9.3 Initial calibration

The GC/MS operating conditions used in our application of the method can be found in Table 2.

9.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 4 for a 5-50 ng injection or purging of 4-bromofluorobenzene. Analyses must not begin until these criteria are met.

a. The BFB mass intensity criteria in Table 4 is used as tuning acceptance criteria. The mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak. Alternatively, the analyst may use an averaged scan across the apex of the peak or other documented approaches suggested by the instrument manufacturer.

NOTE: All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

9.3.2 The sample introduction system conditions used in our application of the method can be found in Table 2. A different calibration curve is necessary for each method because of the differences in conditions and equipment. A minimum of at least five different calibration standards is necessary. Calibration must be performed using the sample introduction technique that will be used for samples. For Method 5030, the purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, develop the standard curve with whichever volume of sample that will be analyzed.

a. Create a calibration curve using standards prepared at the following levels: 0.5, 1, 2, 5, 10, 20, 40 and 50 µg/L. Add 25.0 ± 0.1 mL of water to the 25-mL glass syringe fitted with a luer lock adapter. Next, using a 10-µL microsyringe equipped with a long needle, take a volume (to nearest 1%) of the secondary dilution solution containing appropriate concentrations of the calibration standards. Add the aliquot of calibration solution directly to the water in the syringe by inserting the needle through the luer adapter. When discharging the contents of the microsyringe, be sure that the end of the syringe needle is well beneath the surface of the water. Similarly, add 10 ± 0.05µL of the internal standard/surrogate solution. Carefully discharge the sample into the purge vessel and begin purging.

9.3.3 Proceed with the analysis of the calibration standards following the procedure in the introduction method. Rinse each vessel with water or remove the vessel, rinse and dry for reduction of target analyte carryover between standard analyses.

9.3.4 Tabulate the area response of the characteristic ions (see Table 1) against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured (Sec. 9.6.2).

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The RF is calculated as follows:

$$RF = \frac{A_S \times C_{IS}}{A_{IS} \times C_S}$$

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.
C_{is} = Concentration of the internal standard.

9.3.5 System performance check compounds (SPCCs) - Calculate the mean RF for each target analyte using the five RF values calculated from the initial (5-point) calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane, 1,1-dichloroethane, bromoform, chlorobenzene, and 1,1,2,2-tetrachloroethane. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Example problems include:

a. Chloromethane is the most likely compound to be lost if the purge flow is too fast.

b. Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

c. Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

d. The minimum mean response factors for the volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

9.3.6 Calibration check compounds (CCCs)

a. The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column.

b. Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration, as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}} \quad RSD = \frac{SD}{\overline{RF}} \times 100$$

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where:

RFi = RF for each of the calibration standards
$\overline{RF}$ = mean RF for each compound from the initial calibration
n = Number of calibration standards, e.g., 5

c. The RSD should be less than or equal to 15 % for each target analyte. However, the RSD for each individual Calibration Check Compound (CCC) must be equal to or less than 30%. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

d. If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before re-attempting calibration.

9.3.7 Evaluation of retention times - The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

9.3.8 Linearity of target analytes

a. If the RSD of any target analyte is 15% or less, then the response factor is assumed constant over the calibration range, and the average response factor may be used for quantitation.

b. If the RSD of any target analyte is greater than 15%, use of an additional calibration option is required. A quadratic fit forced to the origin is frequently used for Bromoform and other compounds that tend to be less linear over the calibration range. A linear regression can also be used.

NOTE: Method 8000 specifies a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

9.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

9.4.1 Prior to the analysis of samples or calibration standards, introduce 5-50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 4 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

9.4.2 The initial calibration curve for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 9.4.4 through 9.4.7.

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9.4.3 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples.

9.4.4 System Performance Check Compounds (SPCCs)

a. A system performance check must be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard must meet its minimum response factor (see Sec. 9.3.5.4). This is the same check that is applied during the initial calibration.

b. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

9.4.5 Calibration Check Compounds (CCCs)

a. After the system performance check is met, the CCCs listed in Sec. 9.3.6 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

b. If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift,) for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

c. Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

9.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 sec from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

9.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (- 50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

9.5 GC/MS analysis of samples

9.5.1 Samples can be screened to minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. Sample screening is particularly important when Method

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8260 is used to achieve low detection levels. The most frequently used technique is to analyze a highly diluted sample. Additional screening options available utilizing SW-846 methods are automated headspace-GC/FID (Methods 5021/8015), automated headspace-GC/PID/ELCD (Methods 5021/8021), or waste dilution-GC/PID/ELCD (Methods 3585/8021) using the same type of capillary column. When used only for screening purposes, the quality control requirements in the methods above may be reduced as appropriate.

9.5.2 BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

9.5.3 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice.

9.5.4 The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future analysis. Therefore, if only one VOA vial is provided to the laboratory, the analyst should prepare the remaining sample aliquot for analysis at this time, to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. If the second aliquot is needed, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

9.5.5 Remove the plunger from a 25-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 25.0 mL.

9.5.6 The following procedure may be used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delay, until the diluted sample is in a gas-tight syringe.

a. Dilutions are usually made in the 25-mL syringe. Intermediate dilution steps made in volumetric flasks (10- to 100-mL) may be necessary for extremely large dilutions. Samples may also be diluted in the purge vessel by measuring the sample directly into the purge vessel and adding a volume of organic-free water equaling 25 mL.

b. Measure the volume of organic-free water needed in the 25-mL syringe, and pull the plunger back slightly beyond the 25-mL mark to ensure enough room for the sample.

c. Inject the appropriate volume of the original sample into the syringe.

9.5.7 Compositing aqueous samples prior to GC/MS analysis

a. Add 5 mL of each sample (up to 5 samples are allowed) directly to a purge vessel or to a 25-mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be used, provided that equal volumes of each sample are composited.

b. The samples must be cooled to 4 ºC or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing. Mix each vial well by inverting the vial and/or syringe. Introduce the composited sample into the instrument.

9.5.8 The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 µL of the surrogate spiking solution to 25 mL of aqueous sample will yield a concentration of 10 µg/L of each surrogate standard.

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9.5.9 Add 10 µL of the matrix spike solution (Sec. 8.13) to a 25-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 10 µg/L of each matrix spike standard.

a. Follow the same procedure in preparing the laboratory control sample (LCS), except the spike is added to a clean matrix.

b. If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking and LCS solutions may be required.

9.5.10 Analyze the sample following the procedure in the introduction method of choice.

9.5.11 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion.

a. When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

b. All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

9.5.12 The use of selected ion monitoring (SIM) is acceptable in situations requiring detection limits below the normal range of full EI spectra. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

9.6 Qualitative analysis

9.6.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

a. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

b. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

c. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

d. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is

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achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

e. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

f. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

9.6.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. For example, the RCRA permit or waste de-listing requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:

a. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

b. The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)

c. Molecular ions present in the reference spectrum should be present in the sample spectrum.

d. Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of co-eluting compounds.

e. Ions present in the reference spectrum but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

9.7 Quantitative analysis

9.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of that of a given analyte.

9.7.2 If the RSD of a compound's response factors is 15% or less, then the concentration in the extract may be determined using the average response factor ($\overline{RF}$) from initial calibration data (Sec. 9.3.6). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

9.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 9.6.2) should be estimated. The same formula should be used with the following modifications: The

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areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

9.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

10.0 QUALITY ASSURANCE

10.1 Refer to Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Methods 3500 and 5000. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Section 10.0:

10.2. The GC/MS system must be tuned to meet the BFB specifications in Secs. 9.3.1 and 9.4.1. There must be an initial calibration of the GC/MS system as described in Sec. 9.3. The GC/MS system must meet the SPCC criteria described in Sec. 9.4.4and the CCC criteria in Sec. 9.4.5, each 12 hours.

10.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff is trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

10.4 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). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

10.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

10.4.2 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 expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, a Blank Spike can be used to verify that the laboratory can perform the analysis in a clean matrix.

10.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight

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or volume. The LCS is spiked with the target analytes at a low concentration. The LCS can be used to verify that the laboratory can perform the analysis at or near the detection limit.

10.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

10.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

10.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), re-calibration of the system must take place.

10.7 The laboratory adopts additional quality assurance practices for use with this method, including analyzing standard reference materials and participating in relevant performance evaluation studies.

11.0 METHOD PERFORMANCE

11.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

11.2 This method has been tested using purge-and-trap (Method 5030) in a single laboratory using spiked water. Using a narrow-bore capillary column, water was spiked a concentration of 1 µg/L. Calculated MDLs are presented in Table 3.

12.0 REPORT

12.1 The lab performs a precision and accuracy determination on an annual basis.

12.2 All results should be reported in µg/L unless otherwise specified by the client.

12.3 For waters, one significant figure should be used for reporting results less than 10. Two significant figures should be used for reported concentrations ≥ 10.

13.0 KEY WORDS

8260, Purge and trap, volatile, organic, GC/MS, chromatography, mass spectrometer, calibration

14.0 REFERENCES

14.1 Methods for the Determination of Organic Compounds in Drinking Water, Supplement II Method 524.2; U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring Systems Laboratory, Cincinnati, OH, 1992.

14.2 www.restekcorp.com Technical Guide: “Optimizing the Analysis of Volatile Organic Compounds.”

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14.3 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Method 624," October 26, 1984.

14.4 U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, January 1991.

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TABLE 1:

RETENTION TIMES AND CHARACTERISTIC IONS FOR VOLATILE COMPOUNDS

#	Name	Aprox. Ret Time	Primary ion	Secondary ion(s)
1)	Fluorobenzene	7.66	96	0
2)	Dichlorodifluoromethane	1.66	85	87
3)	Chloromethane	1.85	50	52
4)	Vinyl chloride	1.97	62	64
5)	Bromomethane	2.33	94	96
6)	Chloroethane	2.44	64	66
7)	Trichlorofluoromethane	2.74	101	103
8)	1,1-Dichloroethene	3.38	96	61
9)	Acetone	3.46	43	58
10)	Carbon disulfide	3.67	76	78
11)	Methylene chloride	4.08	84	49
12)	trans-1,2-Dichloroethene	4.50	96	61
13)	Methyl-t-butyl ether	4.53	73	57
14)	1,1-Dichloroethane	5.13	63	65
15)	2,2-Dichloropropane	5.99	77	79
16)	cis-1,2-Dichloroethene	6.00	96	61
17)	2-Butanone	6.05	43	72, 57
18)	Bromochloromethane	6.34	128	49
19)	Chloroform	6.47	83	85
20)	1,1,1-Trichloroethane	6.72	97	99
21)	Carbon tetrachloride	6.96	117	119
22)	1,1-Dichloropropene	6.97	75	110
23)	1,2-Dichloroethene-d4	7.17	65	102
24)	Benzene	7.25	78	0
25)	1,2-Dichloroethane	7.28	62	64
26)	Trichloroethene	8.18	95	130, 132
27)	1,2-Dichloropropane	8.48	63	76
28)	Dibromomethane	8.64	93	95
29)	Bromodichloromethane	8.88	83	85
30)	cis-1,3-Dichloropropene	9.51	75	110
31)	4-Methyl-2-pentanone	9.75	43	58
32)	Toluene-d8	9.89	98	100
33)	Toluene	9.98	92	91
34)	trans-1,3-Dichloropropene	10.30	75	110
35)	1,1,2-Trichloroethane	10.55	97	83
36)	Tetrachloroethene	10.76	166	168
37)	1,3-Dichloropropane	10.79	76	78
38)	2-Hexanone	10.94	43	58, 100
39)	Dibromochloromethane	11.10	129	127
40)	1,2-Dibromoethane	11.25	107	109
41)	Chlorobenzene	11.97	112	77
42)	1,1,1,2-Tetrachloroethane	12.09	131	133, 119
43)	Ethylbenzene	12.14	91	106
44)	m,p-Xylene	12.32	106	91
45)	o-Xylene	12.88	106	91
46)	Styrene	12.90	104	78
47)	1,4-Dichlorobenzene-d4	15.32	152	115

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RETENTION TIMES AND CHARACTERISTIC IONS FOR VOLATILE COMPOUNDS

#	Name	Aprox. Ret Time	Primary ion	Secondary ion(s)
48)	Bromoform	13.15	173	175
49)	Isopropylbenzene	13.42	105	120
50)	Bromofluorobenzene	13.63	95	174
51)	Bromobenzene	13.84	77	156
52)	1,1,2,2-Tetrachloroethane	13.85	83	85
53)	1,2,3-Trichloropropane	13.91	75	77
54)	n-Propylbenzene	14.02	120	91
55)	2-Chlorotoluene	14.14	126	91
56)	4-Chlorotoluene	14.30	126	91
57)	1,3,5-Trimethylbenzene	14.29	105	120
58)	tert-Butylbenzene	14.76	119	91, 134
59)	1,2,4-Trimethylbenzene	14.83	105	120
60)	sec-Butylbenzene	15.08	105	134
61)	1,3-Dichlorobenzene	15.23	146	111, 148
62)	p-Isopropyltoluene	15.30	119	134, 91
63)	1,4-Dichlorobenzene	15.36	146	111, 148
64)	n-Butylbenzene	15.90	91	92, 134
65)	1,2-Dichlorobenzene	15.90	146	111, 148
66)	1,2-Dibromo-3-chloropropane	17.05	75	157, 155
67)	1,2,4-Trichlorobenzene	18.29	180	182, 145
68)	Hexachlorobutadiene	18.56	225	223, 227
69)	Naphthalene	18.64	128	102
70)	1,2,3-Trichlorobenzene	19.01	180	182, 145

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Table 2.

Purge and Trap Conditions

Instrument name	VOL1
P&T	Tekmar 3100 sample concentrator SN# 99196028
Automatic Sampler	Tekmar 2016 P&T autosampler SN# 99042008
Trap	VOCARB 3000
P&T-GC interface	Volatiles interface
Sample size	25 mL
purge temperature	32 C
Purge rate	40 mL/min
Purge time	11 min
Dry purge time	2 min
Desorb preheat	240 C
Desorb temperature	250 C
Desorb time	2 min
Bake temperature	260 C
Bake time	8 min
Bake gas bypass	1 min
Line temperature	135 C
Valve temperature	150 C
Moisture control system:	
Bake temperature	300

Gas Chromatograph and Mass Spectrometer Conditions

Gas chromatograph	**Agilent 6890 SN# US00029052**
Inlet	EPC split/splitless
Mode	Split
Inlet temperature	150 C
Pressure	9.88 psi
Split ratio	30:1
Split flow	38.7 mL/min
Total flow	42.7 mL/min
Oven	
Initial temperature	40 C
Initial time	4 min
rate	10 C/min
Final temperature	220 C
Final time	22 min
Hold time	0 min
Equilibration time	0.25 min
Oven max temperature	250 C
Column	**J&W DB624 122-1334**
Length	30 m
Diameter	253 µm
Film thickness	1.4 µm
Carrier	Helium
Initial flow	1.3 mL/min
Average velocity	41 cm/sec
Mode	Constant flow
Inlet	Back
Outlet	MS
Outlet pressure	Vacuum
Mass Spectrometer	**Agilent 5973 SN# US92522683**
EM Voltage	BFB +/- 200
Solvent delay	none
Low mass	35
High mass	260
Threshold	150
Sampling	3
Scans/sec	3.25
Quad temperature	150
Source temperature	230
Transfer line temperature	230

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Table 3

Compound	mdl
Fluorobenzene	-
Dichlorodifluoromethane	**0.16**
Chloromethane	**0.11**
Vinyl chloride	**0.10**
Bromomethane	**0.13**
Chloroethane	**0.11**
Trichlorofluoromethane	**0.17**
1,1-Dichloroethene	**0.28**
Acetone	**2.09**
Carbon disulfide	**0.38**
Methylene chloride	**0.13**
trans-1,2-Dichloroethene	**0.17**
Methyl-t-butyl ether	**0.16**
1,1-Dichloroethane	**0.12**
2,2-Dichloropropane	**0.16**
cis-1,2-Dichloroethene	**0.16**
2-Butanone	**1.69**
Bromochloromethane	**0.19**
Chloroform	**0.15**
1,1,1-Trichloroethane	**0.15**
Carbon tetrachloride	**0.17**
1,1-Dichloropropene	**0.21**
1,2-Dichloroethene-d4	-
Benzene	**0.13**
1,2-Dichloroethane	**0.14**
Trichloroethene	**0.13**
1,2-Dichloropropane	**0.16**
Dibromomethane	**0.15**
Bromodichloromethane	**0.14**
cis-1,3-Dichloropropene	**0.16**
4-Methyl-2-pentanone	**0.75**
Toluene-d8	-
Toluene	**0.13**
trans-1,3-Dichloropropene	**0.15**
1,1,2-Trichloroethane	**0.22**
Tetrachloroethene	**0.26**
1,3-Dichloropropane	**0.18**
2-Hexanone	**1.08**
Dibromochloromethane	**0.21**
1,2-Dibromoethane	**0.15**
Chlorobenzene	**0.12**
1,1,1,2-Tetrachloroethane	**0.18**
Ethylbenzene	**0.18**
m,p-Xylene	**0.27**
o-Xylene	**0.12**
Styrene	**0.16**
1,4-Dichlorobenzene-d4	-
Bromoform	**0.20**
Isopropylbenzene	**0.16**

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Compound	mdl
Bromofluorobenzene	**0.29**
Bromobenzene	**0.13**
1,1,2,2-Tetrachloroethane	**0.19**
1,2,3-Trichloropropane	**0.16**
n-Propylbenzene	**0.22**
2-Chlorotoluene	**0.19**
4-Chlorotoluene	**0.16**
1,3,5-Trimethylbenzene	**0.19**
tert-Butylbenzene	**0.19**
1,2,4-Trimethylbenzene	**0.16**
sec-Butylbenzene	**0.23**
1,3-Dichlorobenzene	**0.18**
p-Isopropyltoluene	**0.21**
1,4-Dichlorobenzene	**0.16**
n-Butylbenzene	**0.22**
1,2-Dichlorobenzene	**0.14**
1,2-Dibromo-3-chloropropane	**0.33**
1,2,4-Trichlorobenzene	**0.24**
Hexachlorobutadiene	**0.31**
Naphthalene	**0.33**
1,2,3-Trichlorobenzene	**0.18**

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TABLE 4
BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA[a]

m/z	Required Intensity(relative abundance)
50	8 to 40 % of m/z 95
75	30 to 66% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% but less than 120% of m/z 95
175	4 to 9% of m/z 174
176	Greater than 93% but less than101 % of m/z 174
177	5 to 9% of m/z 176

[a] Alternate tuning criteria (CLP) is used.

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W-22

Diesel and Motor Oil Range Compounds, Analysis of

Process Owner (TS/PM/OM/LM)	
Randy Eatherton	
Assignment	Reviewed August 20, 2008 by Christine Devine
Electronic only unless colored "Controlled" stamp is present	

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1.0 SCOPE

1.1 This method adapts EPA SW-846 Methods 3540 and 8000 and covers the quantitative analysis of diesel range compounds in soils.

1.2 The method involves extracting the samples with methylene chloride and injection of a portion into a gas chromatograph equipped with a flame ionization detector (FID). Quantitation is accomplished by integrating to baseline as a group the area of components after dodecane through tetracosane. The sensitivity of the method's compounds depends on the level of interferences in addition to instrumental limitations. Below are the GC limits of detection obtainable in an aqueous matrix in the absence of interferences.

Analyte	Soil	Water (µg/L)
Diesel	25 mg/kg	0.25 mg/L
Motor Oil	100 mg/kg	0.5 mg/L

NOTE: This method can also be extended to heavier hydrocarbon mixtures (e.g., transformer oil, motor oils and Bunker C) with the specific calibration for them as long as the minimum regulatory limits for these mixtures is attained.

1.3 This standard operating procedure follows procedures from Washington State Department of Ecology Analytical Method for Petroleum Hydrocarbons- "Semi-Volatile Petroleum Products Method for Soil and Water," June 1997.

2.0 SUMMARY OF METHOD

2.1 For water analysis, a 500-mL water sample aliquot is spiked with o-terphenyl surrogate. The solution is extracted with methylene chloride and concentrated to 1 mL. An appropriate aliquot is injected in to the gas chromatograph (GC) for separation and detection by a Flame Ionization Detector (FID).

2.2 For soil samples, extraction is completed using sonication per Weyerhaeuser AP E3600 (SOP 4-P-3600.) Approximately 20 g of sample is spiked with o-terphenyl and extracted with methylene chloride. Sample extract is concentrated to a final volume of 10 mL and analyzed using GC/FID.

3.0 INTERFERENCES

3.1 Refer to EPA Method 3500. In addition, avoid contact of samples, equipment, and reagents with any form of plastic or rubber materials including gloves, hand lotion, pH buffers, and ink.

3.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

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 waste to waste, depending upon the nature and diversity of the waste being sampled.

4.0 ESTIMATE OF ANALYTICAL TIME

24 hours for a set of 12 samples, depending on concentration and amount of organic material in the samples. This time limit includes extraction, sample concentration, and GC analysis.

5.0 AMOUNT OF SAMPLE REQUIRED

Water samples are typically 500 mL and 20 g is used for soil samples. Aliquots may vary based on desired detection limits and concentration of analyte.

6.0 SAMPLING, SAMPLE HANDLING, AND PRESERVATION

6.1 Grab samples must be collected in cleaned amber glass bottles with Teflon™-lined lids. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

6.2 Sediment/soil samples: Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

6.3 Waste samples that are dry and amenable to grinding**:** Grind or otherwise subdivide the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. Introduce sufficient sample into the grinding apparatus to yield at least 10 g after grinding.

6.4 Samples must be extracted within 7 days (for water) and 14 days (for soil) after sampling. Extracts must be analyzed within 40 days after the extraction. Both samples and extracts must be stored under refrigeration (at 4 ± 2 ℃.)

6.5 Finished extracts must be stored in 4 ± 2 °C refrigerator until analysis.

7.0 EQUIPMENT REQUIRED

7.1 Glassware

7.1.1 Vacuum flask, 500-mL, Pyrex.

7.1.2 Continuous Liquid/Liquid Extractors.

7.1.3 Volumetric Flasks, 10-mL to 100-mL, Ground-glass Stoppered

7.1.5 Kuderna Danish flask, 500-mL, Pyrex.

7.1.6 Snyder column, three ball, Pyrex.

7.1.7 15-mL concentrator tubes, Kontes.

7.1.8 Beakers, 400mL, Pyrex.

7.2 Gas Chromatography

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7.2.1 Gas Chromatograph - HP 5890 Series II.

7.2.2 Flame Ionization Detector (FID)

7.2.3 Capillary Split/Splitless Injector

7.2.4 J&W Scientific DB-5MS --100 % Methyl Polysiloxane, 30M x 0.25 with 0.25-µm film thickness Capillary Column

7.2.5 Chromatography Data System - hp Enviroquant.

7.3 Other equipment

7.3.1 Gastight Syringe, 10-µL to 100-µL.

7.3.2 Sonicator, Sonics and Materials, Inc.

7.3.3 Analytical Balance, accurate to 0.001 g

7.3.4 Nitrogen evaporator, Organomation model # 112.

7.3.5 Millipore water, organic free.

7.3.6 Glass Wool, Pyrex, hexane rinsed

7.3.7 Teflon boiling stones, rinsed with methylene chloride.

7.3.10 Desiccator.

7.3.11 Aluminum pan, 70 mm, for determining % moisture.

8.0 REAGENTS REQUIRED

All standards are bought as pre-made solutions that have been tested by the manufacture. These solutions are then diluted to the required concentration. The solutions are purchased from Accustandard, Restek and Ultra. Spiking solutions are purchased from different manufactures than the solutions used for standard curves (i.e., the initial calibration standards.) All solutions are purchased with quality control paperwork for the given solutions. Standards are cross- checked by purchasing different standards from different manufactures (i.e., when individual mix A is replaced, a different source is used for the next new individual mix A.)

NOTE: AP Q-StdRefMat for requirements for ordering standards.

WARNING: Many of the compounds used to make up mixed standards are extremely toxic. The practicality of providing hazard evaluation precludes supplying hazard warnings on each substance. Also, the interactive affect of the compounds make providing an overall evaluation impossible.

However, most are at very low concentrations. The standards are also handled in a manner such that the likelihood of contact is very small.

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The greatest **hazard is probably associated with the ability of the solvent used to carry the material through the skin.**

There should be little health risks if handled in accordance with safe working practices.

8.1 Sodium sulfate (granular, anhydrous), Na_2SO_4 – Spread in a shallow baking dish, heat for a minimum of 4 hours at 400-500 °C.

WARNING: Inhalation of dust may cause mild irritation to the respiratory tract.

Eye contact may cause mild irritation and redness, or mechanical irritation.

Skin contact may cause mild irritation.

8.2 Methylene chloride, B & J brand or EM brand.

DANGER: All manipulations involving methylene chloride must be performed within a fume hood or a sealed system to prevent venting into the laboratory. It is non-flammable.

Avoid breathing vapors. Exposure can cause anesthetic or narcotic effects, light-headedness, nausea, vomiting and headache. Excessive exposure may cause irritation to upper respiratory tract Unconsciousness, and death can result from extreme cases of over exposure. High levels may also cause cardiac arrhythmias (irregular heartbeats). Breathing vapors can elevate carboxyhemoglobin levels in the cardiovascular system thereby impairing the blood's ability to transport oxygen. Persons who smoke tobacco products will experience an intensified elevation of carboxyhemoglobin levels. Observations in animals include liver and kidney effects.

Delayed effects: exposure may aggravate symptoms of angina (chest pains). Liver and kidney damage may occur.

Avoid skin contact. Prolonged or repeated exposure may cause skin irritation, even a burn. Repeated contact may cause drying or flaking of skin. Can cause irritation and dermatitis.

Cancer information: Suspect carcinogen. Causes cancer in mice and benign tumors in rats.

Avoid direct sunlight and UV sources. High temperatures and open flame may produce phosgene.

Incompatibility with metals such as: aluminum powders, potassium, sodium, and zinc powder. Avoid unintended contact with amines. Avoid contact with strong bases and strong oxidizers. Avoid prolonged contact with or storage in aluminum or its alloys.

OSHA action level: 12.5 ppm. PEL is 25 ppm. (If you can smell it, the amount is too high.)

8.3 Retention Time Standards. Prepare a composite standard containing, dodecane (C_{12}) and tetracosane (C_{24}) at 25 µg/mL. Weigh 0.25g (± 0.01) of each analyte into a 10-mL volumetric flask and bring to volume with methylene chloride.

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NOTE: See comment under 8.0.

WARNING: See WARNING under 8.0.

NOTE**:** Stock standards required in this method are identical to those required in both WTPH-G and WTPH-HCID.

8.4.1 Once the vials have been opened, transfer the stock standard solutions into bottles with Teflon-lined screw caps. Store at 4 °C and protect from light.

8.4.2 Stock standard solutions must be replaced after 1 year, or sooner if comparison with check standards indicates a problem.

8.4.3 If chemical purity of a compound is 98 % or greater, the mass may be used without correction. If the chemical purity of a compound is less then 98%, then the concentration of the compound needs to be calculated for correct purity.

8.5 Diesel Stock Standard.

8.5.1 Equal mass of #2 diesel oil from at least two different oil companies are mixed together to form a composite diesel fuel. From this composite fuel a stock standard is prepared by adding 0.5 (± 0.001) g of diesel stock to a 100-mL volumetric flask, tared to the nearest 0.001g, containing 60 (± 10) mL of methylene chloride. Re-weigh the flask, then bring to volume with methylene chloride, stopper and mix by inverting the flask several times.

Calculate the concentration as follows:

$$\text{Stock Conc, } \mu\text{g/mL} = \frac{(\text{final mass, mg}) - (\text{tare mass, mg})}{100\ \text{mL}} \times \frac{1000\ \mu\text{g}}{\text{mg}}$$

8.5.2 Another options for the diesel stock standard is to order Diesel #2 at 20,000 µg/mL from two different sources (Ultra, Restek).

NOTE: See comment under 8.0.

WARNING: See WARNING under 8.0.

8.5.3 A second source standard is used for the diesel spike standard. Using the source not used for the calibration, dilute 1 mL (± 10 µL) to 10 mL using a volumetric flask. This should be at a final concentration of 2000 µg/mL.

8.6 Motor Oil Stock Standard. Equal mass of 30 weight motor oil from at least two different oil companies are mixed together to form a composite motor oil. From this composite fuel a stock standard is prepared by adding 0.5 (± 0.001) g of motor oil stock to a 100 mL volumetric flask, tared to the nearest 0.001g, containing 60 (± 10) mL of methylene chloride. Re-weigh the flask, then bring to volume with methylene chloride, stopper and mix by inverting the flask several times.

Calculate the concentration in the same manner as the diesel stock standard.

8.7 Stock Surrogate Standard. The analyst should monitor the performance of the extraction, clean up (when used), and analytical system, and the effectiveness of the method in dealing with each sample matrix, by spiking each sample, standard, and water blank with surrogates. The surrogate

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is o-terphenyl. The use of additional surrogates or different surrogates is optional. The selected surrogate compounds should be non-polar, neutral extractable and observable in a diesel matrix.

8.8 Working Diesel Range Surrogate Spike, 1000 µg/mL. Accurately weigh 100 ± 0.1 mg of o-Terphenyl into a tared 100-mL volumetric flask. Dilute to volume with methylene chloride. Calculate the concentration in the same manner as the diesel standard.

8.9 Calibration Standards.
A calibration solution is made by diluting a stock solution available from Ultra/Restek or made in the lab. Calibration standards, a minimum of 5 concentrations for each analyte of interest, are made in five 10-mL volumetric flask. Spike the flasks with 10 (± 0.2), 20 (± 0.3), 40 (± 0.5), 100 (± 1), and 200 (± 2) µL of the diesel stock and 100 (± 1), 250 (± 2), 500 (± 5), 1000 (± 10) and 2000 (± 10) µL of surrogate spike. The lowest calibration concentrations should be at a concentration near, but above, the practical detection limit (PQL.) The remaining concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC.

NOTE: See comment under 8.0.

WARNING: See WARNING under 8.0.

9.0 PROCEDURE

9.1 Soils

9.1.1 Homogenize the sample and weigh 20 (± 0.1) g of soil into a 400-mL beaker and record to the nearest 0.001g. Add anhydrous Na_2SO_4 until a free-flowing powder is achieved (approximately 20 g.) If it forms a large clump, add more anhydrous Na_2SO_4. Grind to grainy texture and note this in the extraction log. Add 100 (± 1)µL of the 1000 µg/mL surrogate standard solution; 100 ± 10 mL of methylene chloride and sonicate for 3 (± 0.1) min. Decant into a Buchner funnel connecting to a vacuum flask and repeat the solvent/soil sonication 2 more times with new solvent. Each extract is collected into the same vacuum flask.

DANGER: See DANGER under 8.2

9.1.2 Pour the extract into a K-D flask and concentrate as above. Transfer the sample to a centrifulge tube and bring to a final volume of 10 mL (± 100 µL.)

9.1.3 For soil samples, if the % moistures (dry weight) have not been done then a portion of the sample needs to oven dried overnight to determine the % solids as per AM S-2540G.

9.2 Water

9.2.1 Using a Kimax 500-mL graduated cylinder (TD, Class B or better,) measure out a 500 (± 10) mL of sample and transfer it into a 2-L separatory funnel. Record as 500 mL. Spike the sample with surrogate compound(s) according to Part 8.8.

9.2.2 Add 50 mL (± 10) of methylene chloride to separatory funnel and extract the sample by vigorously shaking the funnel for 2-3 min, with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min to a max of 4 hr. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the extractionist must employ mechanical techniques to complete the phase separation.

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The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, warming, or other physical methods.

DANGER: See DANGER under 8.2.

9.2.3 Collect the solvent extract in a 250-mL, ground-glass Erlenmeyer flask.

9.2.4 Repeat the extraction two more times using 50 mL (± 10) of methylene chloride each time. Combine the extracts in the Erlenmeyer flask.

9.2.5 Transfer the extract to a Kuderna-Danish flask connected to a 15-mL concentrator tube. Add one or two Teflon boiling stones and attach a pre-wet Snyder column. Place the KD 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 30-40 min. At the proper rate of distillation the balls of the column will actively chatter, but not flood the chambers with condensed solvent. When the apparent volume of the liquid reaches 2-4 mL, remove the KD apparatus from the steam and allow it to drain and cool for at least 10 min. The solvent in the collector tubes needs to be blown down or refrigerated by the end of the extraction day.

9.2.6 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride and transfer the sample to a centrifuge tube.

9.2.7 Evaporate the solvent to ~0.5-1.0 mL using a gentle stream of clean, dry nitrogen with no water bath. Containers holding samples should be cold and frosty. Transfer the sample to a GC vial and bring to a final volume of 1 ± 0.01 mL.

9.3 GC conditions

Injection sample volume -- 2 µL
Injector temperature -- 290 °C
Ion block temperature -- 300 °C
Hydrogen flow -- 30-35 mL/min
Air flow -- 300-400 mL/min
Carrier flow and Nitrogen make up flow -- 30 mL/min

Temperature program:

Initial temperature -- 50 °C for 2 min
Program rate -- 10 °C /min
Final temperature -- 320 °C for 5 min
Total run time -- 33 min

Inject 2 ± 0.2 µL of the extracts into the GC, utilizing the splitless mode, either manually or by use of an autosampler. Before injecting samples onto the GC, check for dark color to determine if any should require diluting prior to analysis.

Diesel range compounds are indicated if compounds are detected eluting after dodecane (nC_{12}) through tetracosane (nC_{24}).

Motor oil range compounds are indicated by the presence of compounds detected eluting after tetracosane (nC_{24}).

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9.4 Calibration

Refer to AP E-8000 for a description of each of these procedures. Use Table in 2.2 for guidance on selecting the lowest point on the calibration curve.

9.5 Analysis

9.5.1 Refer to AP E-8000.

9.5.2 Follow AP E-8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-concentration standard after each group of 10 samples in the analysis sequence.

9.5.3 Record the sample volume injected and the resulting peak sizes (in peak heights.)

9.5.4 Using the internal calibration procedure (AP E-8000,) determine the identity and quantity of each component peak in the sample chromatogram corresponding to the compounds used for calibration purposes.

9.6 Determine each analyte comparing the area response of the analyte to the average retention factor from the calibration and the area response of the internal standard.

9.6.1 Diesel range compounds. The area of all components after dodecane through tetracosane is integrated to the baseline as a group.

9.6.2 Motor Oil range compounds. For those samples containing compounds eluting after tetracosane, compare the area of the standard to the area, integrated to the baseline, of the compounds.

10.0 QUALITY ASSURANCE

10.1 Quality control to validate sample extraction is covered in AP E-3500 and in the extraction procedure utilized.

10.2 Procedures to check the GC system operation are found in AP E-8000.

10.2.1 Select a representative spike concentration for each compound (diesel or motor oil) to be measured. Using stock standards, prepare a quality control check sample concentrate, in methanol, that is 1000 times more concentrated than the selected concentrations. Use this quality control check sample concentrate to prepare quality control check samples.

10.2.2 A duplicate sample should be extracted and analyzed for every 10 samples. The duplicate should have a % RSD of no more then 10 %.

10.2.3 Each analyst needs to perform the MDL for each matrix and this data should be kept in a training file as per the training SOP.

10.3 Addition of an appropriate surrogate extraction spike to samples and method blank(s) is required. For all samples, blank(s,) duplicates, and/or matrix spikes the recovery of the surrogates should be between 50-150 % and must be reported with the results unless the quantity of product in the extracts precludes the determination.

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If recovery is not within limits, the following procedures are required:

10.3.1 Check to be sure there are no errors in calculations of spike and surrogate solutions. Also, check instrument performance.

10.3.2 Re-calculate the data and/or re-analyze the extract if any of the above checks reveal a problem.

10.3.3 Depending on holding time and sample quantity, the analyst must re-extract and re-analyze the sample if none of the above are a problem or flag the data as "estimated concentration."

11.0 REPORT

11.1 Calculation.

If the RF is < 15 %, then use 11.1.1. If not, use 11.1.2.

11.1.1 For averaged calibrations, the formula is:

$$M_a = \left(\frac{R_a}{RF} \right)$$

Where:

M_a = Mass of Analyte

R_a = Area Response of Analyte

RF = Average Response Factor of analyte

11.1.2 For a linear calibration the formula is:

$$M_a = [m_1 Ra + b]$$

Where:

M_a = Mass of Analyte

m_1 = Slope of calibration

R_a = Area Response of Analyte

b = y- intercept of calibration

11.2 Precision and Accuracy

True precision and accuracy are not applicable to the procedure. There is no reference material to make an appropriate determination for accuracy. Precision may vary among samples due to varying characteristics of the samples, and, for soils, the ability to homogenize them. The nature of the material being measured can also vary.

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11.3 The final concentration is reported in µg/L for water and mg/kg for soil. The unit is determined by using the following calculation:

Final Concentration= $(DF*M_a* FV)/(AI*IVM)$

Where:

DF = Diliution Factor

M_a = Mass of Analyte

FV = Final Volume of Sample

AI = Amount injected

IVM = Initial Volume or mass of Sample

11.4 Report results to two significant figures.

11.5 Detection limits are noted in part 1.2.

12.0 KEY WORDS

AM U-WTPH-D, diesel, diesel range organics, motor oil range organics, oil, motor oil, soil, TPH, water, WTPH

13.0 REFERENCES

Washington State Department of Ecology Analytical Method for Petroeum Hydrocarbons, NWTPH-Dx- Semivolatile Petroleum Products Method for Soil and Water Analysis, Junel 1997.

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W-23

ANALYSIS OF GASOLINE RANGE COMPOUNDS

Process Owner (TS/PM/OM/LM) **Randy Eatherton**	
Assignment	Reviewed July 14, 2003 by Christine Devine Reviewed August 19, 2008 by Christine Devin
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1.0 SCOPE

1.1 The WTPH-G method adapts EPA SW-846 Methods 5030 and 8020 to perform the analysis for gasoline in soils and waters. Soil samples are extracted by shaking in methanol and analyzing 100uL of the methanol diluted into into 5 mL of reagent water. The sample is analyzed utilizing a purge/trap concentrator equipped gas chromatograph with FID detection. Water samples are directly injected into a purge/trap concentrator for analysis.

NOTE: Benzene, toluene, ethyl benzene, and xylenes (BTEX) may be determined simultaneously with gasoline if the requirements of Method 8020 are met (i.e., a PID in series with a FID and the QA/QC associated with 8020).

1.2 The reporting limit for gasoline in soil is 5 mg/kg on a dry-weight basis. The reporting limit for gasoline in water is 0.25 mg/L.

1.3 This standard operating procedure follows procedures from Washington State Department of Ecology Analytical Method for Petroleum Hydrocarbons- "Volatile Petroleum Products Method for Soil and Water" June 1997.

2.0 SUMMARY OF METHOD

2.1 For soil analysis- Soil samples are extracted with methanol using 4-bromofluorobenzene and a,a,a-trifluorotoluene as surrogates. Methanol is mixed with the soil, the extracts are diluted with reagent water and introduced to the purge/trap concentrator. The sample is analyzed by a gas chromatograph with a flame ionization detector (GC/FID).

2.2 For water analysis, a 5-mL water sample is loaded into a gastight syringe and 4-bromofluorobenzene and a,a,a-trifluorotoluene are added as surrogates. The water sample is introduced directly to the purge/trap concentrator and analyzed by a gas chromatograph with a flame ionization detector (GC/FID).

3.0 INTERFERENCES

3.1 Refer to EPA Method 3500. In addition, avoid contact of samples, equipment, and reagents with any form of plastic or rubber materials including gloves, hand lotion, pH buffers and ink.

3.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

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 waste to waste, depending upon the nature and diversity of the waste being sampled.

4.0 ESTIMATE OF ANALYTICAL TIME

Water samples require little preparation and no extraction and therefore only require the time it takes for analysis. A typical batch of 20 samples with QC requires 12 hr. Soil samples require extraction with methanol and require 18 hr for a batch of 20.

5.0 AMOUNT OF SAMPLE REQUIRED

Water samples are typically 5 mL per each analysis. A 40-mL sample size is needed to include QC and possible re-analysis. Soil samples require 5 g per extraction with a total of 50 g to include QC and possible re-extracts. Aliquots may vary based on desired detection limits and concentration of analyte.

6.0 SAMPLING, SAMPLE HANDLING, AND PRESERVATION

Soils samples should be collected in a soil VOA bottle with Teflon-coated, septum-lined tops. They should be filled to the top to minimize headspace above the soil and stored at 4 °C. There is a 14-day holding time for soils and a 7-day holding time for waters, from date sampled to date extracted. If the water sample is preserved with hydrochloric acid (HCl) to a pH < 2 and stored at 4 °C, then the sample has a 14-day holding time from sampling to extraction. If a sample is out of extraction or analysis holding time, or a water sample was not preserved, these facts need to be included in the narrative.

7.0 EQUIPMENT REQUIRED

7.1 Lab Equipment

7.1.1 Gastight Syringe, 10 µL

7.1.2 50-mL Teflon centrifuge tube

7.1.3 Analytical Balance, up to 300 g, accurate to at least 0.001 g

7.1.4 Vortex mixer

7.1.5 Volumetric Flasks, 10-mL to 100-mL, ground glass stoppered

7.1.6 Glass Wool, Pyrex, hexane rinsed

7.1.7 Gastight Syringe, 5mL

7.2 Gas Chromatography

7.2.1 Flame Ionization Detector (FID)

7.2.2 Capillary Split/Splitless Injector

7.2.3 J & W Scientific DB-624 megabore 0.53-mm ID x 30 m, 3-µm film thickness for purge/trap procedure.

7.2.4 Purge/Trap Liquid Concentrator – Tekmar Corporation 4000

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8.0 REAGENTS REQUIRED

8.1 Sodium sulfate (granular, anhydrous), Na_2SO_4 – Spread in a shallow baking dish, heat for a minimum of 4 hr and up to 8 hours at 400-500 °C.

WARNING: Inhalation of dust may cause mild irritation to the respiratory tract.
Eye contact may cause mild irritation and redness, or mechanical irritation.
Skin contact may cause mild irritation.

8.2 Methanol/ Methyl Alcohol, B & J brand or EM brand.

WARNING: All manipulations involving methanol should be performed within a fume hood. Methanol is flammable and burns with a clear, almost invisible flame. It has a flash point of 12 °C, explosive limits in air in the range of 6 - 36 %, and poses a serious fire risk when heated, or exposed to flame or spark (this includes static electricity). Methanol can react vigorously with strong oxidizing materials. Avoid contact with strong acids, acid chlorides, acid anhydrides, and alkali metals.

Avoid breathing vapors. Initial symptoms may be only that of mild intoxication, but may become severe after 12 to 18 hours. Methanol can be irritating to mucous membranes and upper respiratory tract. Exposure can cause headache, muscle weakness, dizziness, nausea, vomiting, narcosis, respiratory failure, low blood pressure, gastrointestinal disturbances, convulsions, and may be fatal. Methanol is a central nervous system depressant and can cause kidney, liver (enlargement), eye (especially optic nerve) and heart damage.

Avoid skin contact. Methanol is absorbed through the skin. Prolonged or repeated skin contact can cause irritation and dermatitis.

Eye contact may cause irritation and/or temporary corneal damage.

Prolonged exposure to methanol has been known to cause fetal development abnormalities in pregnant rats.

8.3 Retention Time Standards. Prepare a composite standard containing, toluene, and naphthalene at 25 µg/mL. Weigh 0.25 ± 0.01 g of each analyte into a 10-mL volumetric flask and bring to volume with methanol, date, and store.

8.4 All standards are bought as pre-made solutions. All solutions are purchased with quality control paperwork for the given solutions. Standards are cross-checked by purchasing different standards from different manufacturers.

NOTE: AP Q-StdRefMat for requirements for ordering standards

8.4.1 Once the vials have been opened, transfer the stock standard solutions into bottles with Teflon-lined screw caps. Date and store at 4 °C and protect from light.

8.42 Stock standard solutions must be replaced after 1 year, or sooner if a comparison with check standards indicates a problem.

8.43 If chemical purity of a compound is 98 % or greater, the mass may be used without correction or computing the concentration of the standard.

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8.5 Gasoline Stock Standards.

8.5.1 Gasoline composite stock standard. The calibration stock standard comes from Accustandard (5000 µg/mL AK101.0 GCS). It is a pre-made composite of gasoline. The calibration stock solution is diluted to 25 µg/mL by dispensing 125 ± 1 µL of calibration stock solution into a 25-mL volumetric flask and taking it up to volume with methanol. Date and store in a Teflon-sealed bottle.

8.5.2 Laboratory Control Spike - The laboratory control spike comes from Ultra scientific (2500 µg/mL RGO-605). The laboratory control spike is diluted to 10 µg/mL by diluting 40 ± 0.5 µL of stock in a 10-mL volumetric flask and taking it to volume with methanol. Date and store in a Telflon-sealed bottle.

8.5.3 The concentrated gasoline stock is used for soil extraction.

8.6 Stock Surrogate Standard

The surrogates are trifluorotoluene (TFT) and bromofluorobenzene (BFB.) Surrogate compounds are non-polar, purgeable from water and observable in a gasoline matrix.

8.6.1 Gasoline Surrogate Stock, 2000 µg/mL. The gasoline surrogate stock comes from Ultra Scientific in methanol. This surrogate stock is also used as the soil surrogate spiking standard (see section 9). Once the vial has been opened, it is transferred to a GC vial and dated.

8.6.2 Working Gasoline Surrogate Spike. Prepare a 20-µg/mL gasoline surrogate spike standard by dispensing 100 uL into a 10-mL volumetric flask and diluting to volume in methanol. Date and store in a Telflon-sealed bottle.

9.0 PROCEDURE

9.1 Soils

9.1.1 Accurately weigh approximately 5 grams, to the nearest 0.001 g, of soil into a 40-mL VOA vial; add 10 ±0.1 µL of the surrogate stock standard and 10 ± 0.1 mL of methanol. Quickly cap the vial and vortex for 1 ± 0.5 min. Allow the soil/methanol mixture to separate, centrifuging if necessary to clarify the methanol extract. For storage, transfer a portion of the extract into a 3.7-mL amber glass GC vial, with a Teflon lined cap, minimizing the headspace, and store in a refrigerator for no longer than one week prior to analysis.

9.1.2 Check to make sure the % solids for the sample have been completed. If not, then a portion of the soil sample needs to be dried to determine the % solids. Follow method AM S-2540G (AM –1 TS-CLP). Spiking of surrogates, extraction and analyses of the quality control samples will be conducted in the same manner as the samples with the exception that no soil is added to the method blank.

9.1.3 A 100 ± 1-µL aliquot of the methanol extract is transferred via a 100-µL gastight syringe to 5 ± 0.2 mL of water in a 5-mL gastight syringe. Immediately inject this water into the purging vessel of the purge and trap device. For samples expected to contain elevated concentrations of gasoline-range volatiles, a smaller aliquot of the methanol extract should be used to remain within the range of the linear calibration curve of the instrument/method.

9.2 Waters

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A 5 ±0.2 mL gastight syringe is filled with sample and inverted to release any air. Add 10 ± 0.1 µL of the working surrogate via a 10-µL gastight syringe to the 5-mL sample. Immediately inject into the purging vessel. For samples expected to contain elevated concentrations of volatile hydrocarbons, a smaller aliquot of sample water should be used in order to remain within the range of the linear calibration curve.

9.3 GC conditions

Injection sample volume – 5-mL purge and trap Injector temperature - 240 °C Hydrogen flow - 30-35 mL/min Air flow - 300-400 mL/min Carrier flow and Nitrogen make up flow - 30 mL/min	Temperature program: Initial temperature - 30 °C for 5 min Program rate A - 10 °C /min Final temperature A - 180 °C for 5 min Program rate B - 30 °C/min Final temperature B - 220 °C for 2 min Total run time - 28 min

9.4 Purge and Trap operating parameters.

Purge gas	Helium	Desorb temp.	180 °C
Purge time	11.0 min	Bake time	10 min
Purge temp.	Ambient	Bake temp.	180 °C
Desorb time	1.5 min		

Gasoline range compounds are indicated if compounds are detected eluting after toluene through dodecane.

9.5 Calibration

9.5.1 Follow the procedure outlined in EPA Method 8020 for the set up of the purge and trap operating parameters and for the GC. Adjust the hydrogen/air flow rates to optimize the FID sensitivity.

9.5.2 The aqueous purge gasoline/surrogate standards are each prepared by adding 10 ± 0.1 µL, 15 ± 0.2 µL, 20 ± 0.2 µL, 50 ± 1 µL and 100 ± 5 µL of the secondary dilution standard to 5 mL of water. This is done by injecting each aliquot into the end of the 5-mL gastight syringe containing 5 ± 0.2 mL of organic-free water. The calibration standard quantities in the purged water are then 250 ng, 375 ng, 500 ng, 1000 ng, and 2500 ng for gasoline and 50 ng, 100 ng, 250 ng, 500 ng, and 1000 ng for each surrogate compound. Extending the calibration range is allowed as long as the ratio of gasoline to surrogate is retained and the concentrations remain within the linear range of the instrument.

9.5.3 Refer to AP E-8000.

9.5.4 Follow AP E-8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-concentration standard after each group of 10 samples in the analysis sequence.

9.5.5 Record the sample volume injected and the resulting peak sizes (in peak heights).

9.5.6 Using the internal calibration procedure (AP E-8000), determine the identity and quantity of each component peak in the sample chromatogram corresponding to the compounds used for calibration purposes.

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9.5.7 Calculation of a calibration factor may be used for quantitation if the RSD of the individual calibration factors is $\leq$ 20 %. Daily standards (mid-range) are to be run prior to sample analyses and as the last analysis of the day, and continuing calibration analyses (mid-range) should be performed after every 10 samples. The values obtained for these standard analyses must be between ± 15 % of the known value. Should the value of these standards fall outside the ± 15 % limits, then a new five-point calibration must be run.

9.5.8 Should the sample analyzed show concentrations of gasoline above the calibration curve, a reagent water blank should be analyzed to ensure that no "carry-over" will be experienced in subsequent analyses.

10.0 QUALITY ASSURANCE

10.1 Along with each sample set, run at least one sample duplicate per 10 samples (10 % QC) and for each extraction day, run at least one method blank (5 % QC). An instrument blank needs to bracket the standards and a retention check should be analyzed at least every 10 analysis days to determine if any retention-time drift has occurred.

10.2 Select a representative sample for adding spikes. Using stock standards, prepare a quality control check sample concentrate, in methanol, that is 1000 times more concentrated than the selected concentrations. Use this quality control check sample concentrate to prepare quality control check samples.

10.3 A duplicate sample should be extracted and analyzed for every 10 samples. The duplicate should have a % RSD of no more than 10 %.

10.4 Each analyst needs to perform an IPAR for each matrix and this data should be kept in a training file as per the training SOP.

10.5 Addition of an appropriate surrogate extraction spike to samples and method blank(s) is required. For all samples, blank(s), duplicates and/or matrix spikes, the recovery of the surrogates should be between 50-150 % and must be reported with the results. If the surrogate recovery can not be calculated due to a high level of gasoline contamination, then this fact needs to be reported.

10.6 If recovery of the gasoline-range or the surrogates is not within limits, the following procedures are required:

Check to be sure there are no errors in calculations of spike and surrogate solutions. Also, check instrument performance. Re-calculate the data and/or re-analyze the extract if any of the above checks reveal a problem. Re-extract and re-analyze the sample if none of the above are a problem or flag the data as "estimated concentration."

11.0 REPORT

11.1 Gasoline Range compounds.

The area of all components after toluene through naphthalene is integrated to the baseline as a group.

11.2 CALCULATION

11.2.1

For averaged calibrations the formula is:	For a linear calibration the formula is:

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$M_a = \left(\frac{R_a}{\overline{RF}}\right)$ Where: M_a = Mass of Analyte R_a = Area Response of Analyte $\overline{RF}$ = Average Response Factor of analyte	$M_a = \lfloor m_1 Ra + b \rfloor$ Where: M_a = Mass of Analyte m_1 = Slope of calibration R_a = Area Response of Analyte b = y - intercept of calibration

11.2.2 Soils. The area of the components (from toluene through dodecane) is integrated as a group to the baseline and compared to concentrations of the gasoline standards, which are integrated in the same manner. Sample concentrations are to be reported on a dry weight basis.

$$\text{Soil Sample Conc., mg/kg} = \frac{(A \times R) \times D}{E \times M \times S}$$

Where:

A = group area of sample
R = Response factor from standard curve (ng purged/area count)
D = Extract Volume, mL
M = Mass of sample, g
E = Volume of extract purged or injected, µL
S = Decimal percent solids of sample

11.2.3 Waters. The area of the components (from toluene through dodecane) is integrated as a group to the baseline and compared to concentrations of the gasoline standards, which are integrated in the same manner.

$$\text{Water Sample Conc., µg/L} = \frac{(A \times R)}{V}$$

Where

A = group area of sample
R = Response factor from standard curve (ng purged/area count)
V = Volume of sample purged, mL

11.3 PRECISION AND ACCURACY- See Table 1 below for laboratory mean and standard deviation for this method.

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Table 1- Accuracy and Precision for Gasoline Range Organics and Surrogates in Organic-Free Reagent Water Matrix

Compound	Spike Concentration (ug/mL)	Mean Percent Recovery	Standard Deviation of Percent Recovery
Gasoline Range Organics	1	93	10.3
4-Bromofluorobenzene	0.2	99	10.3
a,a,a-Trifluorotoluene	0.2	100	11.1

11.4 Report results, mg/kg for solids and mg/L for liquids, to two significant figures. Low-level results are reported in mg/kg for solids and mg/L for liquids, and are reported to three significant figures.

11.5 Reporting limits are noted under part 1.2.

11.6 Laboratory minimum detection limits for gasoline are 0.00444mg/L for aqueous samples.

12.0 KEY WORDS

13.0 REFERENCES

13.1 Washington State Department of Ecology Analytical Method for Petroleum Hydrocarbons, NWTPH-Gx- Volatile Petroleum Products Method for Soil and Water Analysis, June 1997.

13.2 EPA Method 602, 40CFR136.

13.3 EPA RCRA SW-846 Methods 5030 and 8020.

13.4 State of Oregon Department of Environmental Quality Method TPH-G.

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W-24

Cyanide, Total

Process Owner (TS/PM/OM/LM)	
Maxine Ranta	
Assignment	Reviewed February 24, 2006 by Christine Devine Reviewed August 27, 2008 by Christine Devine
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1.0 SCOPE

1.1 This method is applicable to the determination of cyanide in drinking, surface and saline waters, domestic and industrial wastes, soil and sludges.

1.2 Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

1.3 The working range of the spectrophotometric method is 0.01 to 0.2 mg/L for waters and 0.50 to 10 mg/kg for solids. Higher level samples must be diluted to fall within the working range.

1.4 This method is based upon EPA 335.2 CLP-M, USEPA Contract Laboratory Program, SOW No. 788, rev. Feb.1989.

2.0 SUMMARY OF METHOD

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 colorimetric measurement, the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on 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.

3.0 INTERFERENCES

3.1 Interferences are eliminated or reduced by using the distillation procedure described in section 9.2.

3.2 Sulfides

Sulfides adversely affect the colorimetric procedure. If a drop of the distillate on lead acetate test paper indicates the presence of sulfides, treat 25 mL more of the sample than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to be used for analysis. Avoid a large excess of cadmium and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Sulfides should be removed prior to preservation with sodium hydroxide as described in 6.3.

3.3 The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 antifoam agent will prevent the foam from collecting in the condenser. Fatty acids will distill and form soaps under alkaline conditions, making the end point of titration almost impossible to detect. When this occurs, one of the spectrophotometric methods should be used (Section 2.2).

3.4 Oxidizing agents such as chlorine decompose most of the cyanides. The sample should be tested in the field for the presence of oxidizing agents.

4.0 ESTIMATE OF ANALYTICAL TIME

4.1 Single sample – 2.5 hours.

4.2 Best batch of 10 - 6 hours.

5.0 AMOUNT OF SAMPLE REQUIRED

A minium of 50 mL of sample is required for a water sample. If QC is to be performed, 500 mL of sample should be collected. Solid samples require the equilivant of 1.0g of OD sample to perform the analysis. Extra sample is required to perform a solids determination. If QC is to be performed at least 10g of sample should be collected.

6.0 SAMPLING, SAMPLE HANDLING AND PRESERVATION

6.1 The liquid samples should be collected in plastic 500-mL bottles. All bottles must be thoroughly cleansed and thoroughly rinsed to remove soluble material from containers.

6.2 Oxidizing agents such as chlorine decompose most of the 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 an additional 0.6 g of ascorbic acid for each liter of sample volume.

6.3 Samples must be preserved with 2 mL of 10 N sodium hydroxide per liter of sample (pH ≥ 12) at the time of collection. If storage is required, the samples should be stored at 4º ± 2℃. Holding time is 14 days.

6.4 Solid samples should be collected in wide mouth 8-oz soil jars. No preserative is added.

6.5 Solid samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored at 4º ± 2℃. Holding time is 14 days.

7.0 EQUIPMENT REQUIRED

7.1 Reflux distillation apparatus-round bottom boiling flask (100mL), inlet tube, side-arm, condenser, impinger. See figure 1.

7.2 Graduated test tubes - 50-mL

7.3 Spectrophotometer suitable for measurements at 578 nm with a 1-m cell.

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8.0 REAGENTS REQUIRED

All reagents must be labeled, dated and initialed. Record this data in the appropriate laboratory notebook.

8.1 **Sodium hydroxide solution - 1.25 N:** Dissolve 50 g of NaOH in water, cool to room temperature and dilute to 1 liter with water. Mix well. **See danger under Appendix A section 1.1.**

8.2 **Sulfuric Acid, 1:1:** Add 200mL of water to a 500-mL Erlenmeyer flask. Cool in an ice bath placed on a stirrer. While stirring constantly, slowly and carefull add 200 mL of concentrated H_2SO_4. Allow to cool to room temperature and transfer to a stoppered bottle. **See danger under Appendix A section 1.2.**

DANGER: Too fast addition of acid can cause superheating of the water triggering a violent eruption of material out of the flask. Wear protective clothing, i.e., face shield, acid resistant gloves, and plastic apron. If acid should come into contact with skin, flush with large amounts of water.

8.3 **Cadmium carbonate - powdered: See caution under Appendix A section 1.3.**

8.4 **Ascorbic acid, crystals**

8.5 **Dilute sodium hydroxide solution, 0.25 N:** Dilute 200 mL of sodium hydroxide solution (8.1) to 1000 mL with water. Mix well. **See danger under Appendix A section 1.1.**

8.6 **Sodium dihydrogenphosphate, 1 M**: Dissolve 34.5 g of $NaH_2PO_4 \bullet H_2O$ in 250 mL of water. Mix well. Refrigerate this solution. **See danger under Appendix A section 1.4.**

8.7 **Stock Cyanide Solution – 1000µg/mL:** Dissolve 0.6275 g of KCN and 0.5 g KOH in a 250-mL volumetric flask with water. Mix well. Standardize daily with 0.02 N $AgNO_3$. See steps 12.1-12.3. Dilute to appropriate concentration so that 1 mL = 1 mg CN. **See danger under Appendix A section 1.5.**

8.8 **Working Cyanide Solution – 5.0µg/mL:** Prepare fresh daily by diluting 0.5 mL of stock cyanide solution to 100 mL in a volumetric flask with water. Mix well. 1 mL = 5.0 µg CN (5.0 µg/mL). **See danger under Appendix A section 1.5.**

8.9 **Working Cyanide Solution – 0.5µg/mL**: Prepare fresh daily by diluting 1.0 mL of 5.0 µg/mL cyanide working standard to 10 mL with water. Mix well. **See danger under Appendix A section 1.5.**

8.10 **ICV Cyanide Solution – 1000µg/mL:** Dissolve 0.6275 g of KCN (must be a different lot from the stock cyanide used to prepare the curve) and 0.5 g KOH in a 250-mL volumetric flask with water. Mix well. Standardize daily with 0.02 N $AgNO_3$. See steps 12.1-12.3. Dilute to appropriate concentration so that 1 mL = 1 mg CN. **See danger under Appendix A section 1.5.**

8.11 **Standard silver nitrate solution, 0.10 N:** Prepare from Dilut-it using water. **See warning under Appendix A section 1.7.**

8.12 **Silver nitrate solution 0.02 N:** Aliquot 20 mL of 0.10N silver nitrate (8.11) into a 100-mL volumetric flask. Dilute to volume with water. Mix well. (0.96 mL = 1 mg CN). **See warning under Appendix A section 1.7.**

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8.13 **Rhodanine Indicator**: Dissolve 20 mg of p-dimethyl-amino-benzalrhodanine in 100-mL volumetric flask with acetone. Mix well.

8.14 **Chloramine-T solution**: Dissolve 1.0 g of white, water-soluble Chloramine-T in 100-mL volumetric flask with water. Mix well and refrigerate until ready to use. Prepare fresh weekly. **See warning under Appendix A section 1.8.**

8.15 **Color Reagent**: Pyridine-Barbituric Acid Reagent: Place 6 g of barbituric acid in a 100-mL volumetric flask and add enough water to wash the sides of the flask and wet the barbituric acid. Add 30 mL of pyridine and 6 mL of concentrated HCl. Add a stir bar and water to bring almost to final volume, mix, and cool to room temperature. Remove the stir bar. Dilute to 100 mL with water and mix. This reagent is stable for approximately six months if stored in a cool, dark place. **See warning under Appendix A section 1.9. See caution under Appendix A section 1.10.**

8.16 **Magnesium chloride solution**: Weigh 255 g of $MgCl_2 \bullet 6H_2O$ into a 500-mL volumetric flask, dissolve and dilute to volume with water. Mix well. **See caution under Appendix A section 1.11.**

9.0 PROCEDURE

9.1 Standard Curve

9.1.1 Standards require the same caustic concentration as the samples. Add 5 mL of 1.25 N NaOH to the standard tubes along with water to obtain a volume of 25 mL.

Std Conc. in µg/mL	mL of 0.5 µg/mL (8.9) working std. to 25 mL	mL of 5 µg/mL (8.8)working std. to 25 mL
0	0.0	
0.01	0.5	
0.02	1.0	
0.05		0.25
0.10		0.50
0.20		1.0

9.1.2 Develop the color on the standards starting with step 9.3.2.

9.2 Sample Distillation

9.2.1 Place 50 mL of a liquid sample, or an aliquot, diluted to 50 mL in the 100-mL boiling flask. For a solid sample weigh the equvilant of 1.0g ± 0.001 oven dried material. Wash this into the 100-mL boiling flask using approximately 50 mL of water. Add 10 mL 1.25 N NaOH and approximately 10 mL H_2O to the absorbing tube to obtain an adequate depth of liquid in the absorber. Connect the inlet tube, boiling flask, condenser, impinger and absorber.

9.2.2 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube.
Make sure that the inlet tube is below the level of the liquid and that bubbles are rising through the sample. If not, there is a leak in the apparatus, which MUST be fixed before the acid addition in step 9.2.3.

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CAUTION: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.

9.2.3 Slowly pipet 5 mL 1:1 sulfuric acid (8.2) into the air inlet tube. Allow the airflow to mix the flask contents for approximately 3 min. Pipet 2 mL $MgCl_2$ (8.16) into the air inlet and wash down with a stream of water.

9.2.4 Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing from the air inlet tube. Reflux for 90 minutes. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

9.2.5 Remove the impinger from the absorber, rinsing the inside and outside of the impinger into the absorber. Quantitatively transfer the sample to a large test tube (Pyrex, no. 9826) that has a 50-mL mark etched into the glass. Bring to final volume with water to mark on test tube. Cap the test tube and mix, inverting completely 3 times.

9.3 Sample Analysis

9.3.1 Use half of the sample amount for colorimetric determination, holding the remainder in reserve for dilutions if necessary. Pipet 25 mL of sample into a Pyrex test tube with a 50-mL mark.

9.3.2 Add 7.5 mL of the sodium dihydrogen phosphate buffer solution (8.6) to the sample and mix using a vortex mixer.

9.3.3 Add 1 mL Chloramine-T solution (8.14) to the sample and mix using a vortex mixer. Set timer and allow to stand for 2 min.

9.3.4 Add 2.5 mL of pyridine-barbituric acid solution (8.15) with a glass pipet and mix using a vortex mixer.

9.3.5 Dilute to 50-mL mark, cap and mix by inverting. Set timer and allow to stand for 8 minutes for full color development. Do a visual examination of the color, comparing the hue of the sample to that of the standards, to see if the samples will fall within the curve. If so, continue with the duplicate and spike samples in the same manner. If not, the appropriate samples will need to be diluted as well as the duplicate and spike samples if they fall outside of the curve.

9.3.6 Read the standards and samples on the spectrophotometer at 578 nm. Record the absorbance and concentration.

10.0 CYANIDE STANDARDIZATION

10.1 Fill a 10-mL burette with 0.02N $AgNO_3$ (8.12). Pipet 10 mL of the 1000 mg/L cyanide stock standard (8.7) into a 30-mL beaker. Add one drop of rhodanine indicator (8.13). Titrate approximately 9.6 mL (drop wise after dispensing 9 mL) into the yellow solution until the pink endpoint is reached.

10.2 Repeat for the 1000 ppm ICV standard (8.10).
10.3 Record end points in mL on the AME-335.2- CLP- M standardization sheet.

11.0 QUALITY ASSURANCE

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11.1 Spikes are run on every 20th sample and duplicates on every 10th sample, or one per service request, whichever is more frequent.

11.2 Normally expected QC parameters are as follows:

11.2.1 Calibration Curve – Coefficient of Correlation (r) 0.995

11.2.2 ICV and CCV Recovery 100 ± 10%

11.2.3 ICB and CCB < 0.01 mg/L

11.2.4 Spike Recovery 100 ± 25%

11.2.5 Duplicate RPD 20 %

12.0 REPORT

12.1 Calculations

12.1.1 Record absorbance readings and cyanide concentration from the curve (mg/L) onto AM E- 335.2 M data sheet. Calculations are as follows:

12.1.2 Water:

$$\text{Cyanide mg/L} = \frac{(\text{mg cyanide/L from curve})\,(50\text{mL})\,(\text{final volume mL})}{(\text{mL sample})\,(\text{aliquot used mL})}$$

12.1.3 Soil:

$$\text{Cyanide mg/Kg} = \frac{(\text{mg cyanide/L from curve})(50\text{ mL})(\text{final volume mL})}{(\text{gm sample, OD})(\text{aliquot used mL})}$$

12.2 Report final value to two decimal places with no more than three significant figures.

12.3 Precision and Accuracy

From the most recent 19 runs, expected precision of 4.7% RSD of a 0.10 mg/L standard and an expected accuracy of 97.6 ± 4.7% recovery for the same standard. For the year 2005 a 0.02 mg/L standard gave an IDL of 0.003. This gives a PQL of 0.009.

13.0 KEY WORDS

EPA 335.2, cyanide, water, wastewater, soil

14.0 REFERENCES

14.1 "Standard Methods for the Examination of Water and Wastewater," 20th Edition, 1998.

Appendix A

1.1 Sodium hydroxide

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DANGER: Degree of hazard of sodium hydroxide depends upon form (solid, liquid) or concentration.

Handle with care. Wear safety goggles and a plastic apron. Sodium hydroxide is a strong, corrosive base and the solid or liquid forms can cause severe burns. Carefully add caustic to water with frequent or continuous stirring. Mix in a tub or stoppered sink. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.
Harmful if inhaled. Effects from inhalation of dust or mist (in such cases, use in a hood) vary from mild irritation to serious damage to tissue of the mucous membranes and upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Severe pneumonitis may occur.
Inhalation may result in spasm, inflammation and edema of the larynx and bronchi, chemical pneumonitis and pulmonary edema. Symptoms of exposure may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting.
Skin contact can cause severe irritation or severe burns and scarring with greater exposures. Nitrile gloves should be worn.
Eye contact can cause irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision or blindness.
Very hygroscopic. Can slowly pick up moisture from air and react with carbon dioxide from air to form sodium carbonate.
Sodium hydroxide in contact with strong acids and organic halogen and chloro-organic compounds, especially trichloroethylene, may cause violent reactions. Contact with nitromethane and other similar nitro compounds causes formation of shock-sensitive salts. Contact with metals such as aluminum, magnesium, tin, and zinc cause formation of flammable/explosive hydrogen gas. Avoid peroxides, flammable liquids, and chloroform/methanol mixtures.
Sodium hydroxide, even in fairly dilute solution, reacts readily with various sugars to produce carbon monoxide.
Can react with certain metals, such as aluminum, to generate flammable hydrogen gas.
Permissible exposure limit (PEL) for dust is 2 mg/m3 (Ceiling).

1.2 Sulfuric Acid

DANGER: Degree of hazard of sulfuric acid depends upon concentration. Conc. acid mainly refers to those over 52 %.

Handle with care. Wear safety goggles and a plastic apron. Use in a hood if mist or fumes generated. Conc. sulfuric acid is a strong, corrosive acid and can cause severe burns. Very carefully add acid to water with frequent or continuous stirring. Mix in a tub or stoppered sink. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.
Conc. sulfuric acid is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes and skin.
Inhalation of mist or heated fumes may result in spasm, inflammation and edema of the larynx and bronchi, chemical pneumonitis and pulmonary edema. Symptoms of exposure may include burning sensation or irritation of the nose and throat, coughing, wheezing, laryngitis, shortness of breath, labored breathing, headache, nausea and vomiting.
Avoid skin contact. Laminated film gloves should be worn. Conc. acid is extremely corrosive. Symptoms of redness, pain, and severe burn can occur. Circulatory collapse with clammy skin, weak and rapid pulse, shallow respirations, and scanty urine may follow skin contact or ingestion. Circulatory shock is often the immediate cause of death.
Avoid eye contact. Extremely corrosive. Sulfuric acid can cause severe irritation or burns and result in permanent damage. Contact can cause blurred vision, redness, pain and severe tissue burns. Strong concentrations will probably cause blindness.
Long-term exposure may cause erosion of teeth.

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Sulfuric acid is an oxidizer and strong dehydrating agent. May cause ignition of finely divided organic materials on contact. Reacts with most metals to produce hydrogen gas, which can form an explosive mixture with air. Keep away from bases, carbides, chlorates, fulminates, nitrates, picrates, cyanides, alkali halides, zinc iodide, permanganates, hydrogen peroxide, azides, perchlorates, nitromethane, phosphorous, nitrites, halogens, metal acetylides, oxides and hydrides, metals (especially finely powdered ones) (yields hydrogen gas), strong oxidizing and reducing agents, water, and many other reactive substances.
Harmful if absorbed through skin.
Reaction products include hydrogen, from reaction with metals, or sulfur oxide fumes when heated.
Permissible exposure limit (PEL) is 1 mg/m3.
Mist is a known carcinogen, depending on duration and level of exposure.

1.3 Cadmium carbonate

CAUTION: Cadmium carbonate dust is an irritant. Avoid contact with eyes and skin. Harmful if inhaled or ingested. Material should be handled or transferred in an approved fume hood or with adequate ventilation. Protective gloves (nitrile) should be worn to prevent skin contact. Safety glasses with side shields should be worn at all times.

Tests on laboratory animals indicate material may produce adverse mutagenic effects. Cited in Registry of Toxic Effects of Chemical Substances (RTECS)
OSHA – PEL: TWA – 0.005 CD
ACGIH – TLV: TWA – 0.01 CD

1.4 Sodium dehydrogenate phosphate, monobasic, monohydrate

WARNING: Avoid breathing dust. Dibasic sodium phosphate may cause irritation to the respiratory tract. Symptoms may include coughing and shortness of breath.

Avoid skin and eye contact: May cause irritation with redness and pain.
Incompatible with acids, alkaloids, lead acetate, antipyrine, chloral hydrate, resorcinol and pyrogallol.

1.5 Potassium cyanide

DANGER: Poison. Corrosive on contact with moisture. Causes severe skin and eye burns. Reacts with water or any acid releasing hydrogen cyanide. May be fatal if swallowed, inhaled, or absorbed through skin. Hygroscopic. Exceptional health and contact hazards. Goggles, lab coat and neoprene gloves should be worn. Use in a properly ventilated fume hood.

Avoid skin contact. Will cause severe irritation and burns. Skin absorption may prove fatal. Neoprene gloves are recommended. Flush with water for at least 15 minutes.
Avoid eye contact. Will cause severe irritation and burns. May cause permanent eye damage.
Inhalation: cyanosis, unconsciousness, headache, nausea, vomiting, dizziness, weakness, rapid ineffective breathing, low blood pressure, convulsions and may be fatal.
Ingestation: cyanosis, unconsciousness, headache, nausea, vomiting, dizziness, weakness, rapid ineffective breathing, low blood pressure, convulsions and may be fatal.

NOTE: Keep cyanide antidote kit on hand at all times! If unconscious, break an amyl nitrite pearl in a cloth and hold lightly under nose for 15 seconds. Repeat 5 times at about 15 second intervals. If not breathing, give artificial respiration. Give oxygen.

Target organs: cardiovascular system, central nervous system, liver, kidneys, skin.
Medical conditions generally aggravated by exposure: central nervous system disorders, kidney disorders, respiratory system disease, thyroid disorders, heart disorders, damaged skin.
Store in dry area away from acids and carbon dioxide in light-resistant containers. Contact with water liberates toxic gas. Contact with acids liberates very toxic gas. Do not empty into drains.

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TLV/TWA: 5 mg/m^3
PEL: 5 mg/m3. PEL is for cyanides and CN.

1.6 Potassium hydroxide

DANGER: Degree of hazard depends upon form (solid, liquid) or concentration. Skin contact with the solid hydroxides or concentrated solutions can cause rapid tissue destruction and severe burns. Handle with care. Wear safety goggles and a plastic apron. Potassium hydroxide is a strong, corrosive base and the solid or liquid forms can cause severe burns. Carefully add caustic to water with frequent or continuous stirring. Mix in a tub or stoppered sink. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.

Harmful if inhaled. Effects from inhalation of dust or mist (in such cases, use in a hood) vary from mild irritation to severe damage to tissue of the mucous membranes and upper respiratory tract, depending on severity of exposure. Symptoms may include coughing, sneezing, sore throat or runny nose, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Inhalation may result in spasm, inflammation and edema of the larynx and bronchi, chemical pneumonitis and pulmonary edema. Symptoms of exposure may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting.
Skin contact can cause severe irritation or severe burns and scarring with greater exposures. Prolonged contact with dilute solutions or dust of potassium hydroxide has a destructive effect on tissue.
Eye contact can cause irritation of eyes, with tearing, redness, and swelling, and with greater exposures it can cause burns that may result in permanent impairment of vision or blindness.
Very hygroscopic. Can slowly pick up moisture from air and react with carbon dioxide from air to form postassium carbonate.
Potassium hydroxide in contact with water, flammable liquids, strong acids and organic halogen, especially trichloroethylene, may cause fire or explosion. Contact with nitromethane and other similar nitro compounds causes formation of shock-sensitive salts. Contact with metals such as aluminum, magnesium, tin, and zinc cause formation of flammable/explosive hydrogen gas. Avoid copper and organic material.
Permissible exposure limit (PEL) for dust is 2 mg/m3 (Ceiling.)

1.7 Silver nitrate

DANGER: Poison. Corrosive. Causes burns to any area of contact. May be fatal if swallowed. Harmful if inhaled. Strong oxidizer. Contact with other material may cause fire.

<u>Effects of overexposure</u>
Inhalation: Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, and laryngitis, shortness of breath, headache, nausea and vomiting. May be absorbed into the body following inhalation with symptoms paralleling those from ingestion exposure. Dust deposits in the lungs may resemble a form of pneumoconiosis.
Ingestion: Corrosive. Swallowing can cause severe burns of the mouth, throat, and stomach. Can cause sore throat, vomiting, diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death.
Skin contact: Corrosive. Symptoms of redness, pain and severe burn can occur. Any protective gloves will be sufficient.
Eye contact: Corrosive. Can cause blurred vision, redness, pain, severe tissue burns and eye damage.
Chronic exposure: Repeated application of ingestion causes a permanent bluish discoloration of the skin, conjunctiva, and mucous membranes. Repeated inhalation may cause lung disease.
PEL: 0.01 mg/m3 for silver metal dust and fume as Ag.

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ACGIH – TLV: 0.01 mg/m3 for soluble silver compounds as Ag.
Store in tightly closed container in cool dry place. Incompatibles are ammonia, alkalis, antimony salts, arsenites, bromides, carbonates, chlorides, iodides, thiocyanates, ferrous salts, phosphates, tannic acid and tartrates.

1.8 Chloramine-T

WARNING: Harmful if swallowed or inhaled. Causes irritation to skin, eyes and respiratory tract. May cause allergic skin or respiratory reaction.

Potential health effects
Inhalation: can cause irritation of mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, sore throat, bronchitis, pneumonitis, and possible pulmonary edema. May produce pulmonary sensitization or allergic asthma.
Ingestion: Causes irritation to the gastrointestinal tract. Symptoms may include nausea, vomiting and diarrhea.
Skin contact: Causes irritation to skin. Symptoms include redness, itching, and pain. May cause allergic skin reactions. Any protective gloves will be sufficient.
Eye contact: Causes irritation, redness and pain.
Store at room temp. Incompatibles are strong oxidizers, strong acids, ammonia, and ammonium salts.

1.9 Pyridine

DANGER: Harmful or fatal if swallowed. Harmful if inhaled or absorbed through skin. Affects central nervous system, liver and kidneys. Flammable liquid and vapor. Causes severe irritation to eyes, skin and respiratory tract.

Potential health effects
Inhalation: Causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, shortness of breath, coughing, insomnia, diarrhea, gastrointestinal disturbances, and back pain with urinary frequency. Liver and kidney damage may occur. May be fatal.
Ingestion: Toxic effects parallel those of inhalation. Oral doses of several ounces have been fatal to humans.
Skin contact: Causes severe irritation, possibly burns to the skin. Wear laminated film or PVA gloves. Symptoms include redness and severe pain. Absorption through the sin may occur, resulting in toxic effects similar to inhalation. May act as a photo sensitizer.
Eye contact: Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Chronic exposure: Liver and kidney damage has been reported.
Persons with pre-existing skin, eye or central nervous system disorders, or impaired liver, kidney, or pulmonary function may be more susceptible to the effects of this substance.
Flammable: Flash point- 20ºC. Liquid contact with strong oxidizers may cause fire.
OSHA: PEL-TWA: 5 ppm
ACGIH: TLV – TWA: 5 ppm
NIOSH: PEL: 5 ppm
Incompatible with heat, flame, maleic anhydride, perchromates, strong acids, strong oxidizers. Will attack some forms of plastics, rubber and coatings.

1.10 Barbituric acid

Caution: Irritant. Irritating to eyes, respiratory system and skin. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Protective apparel are recommended.

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1.11 Magnesium chloride

CAUTION: Inhalation of dust may cause mild irritation to the mucous membranes.
Ingestion: Since magnesium salts are slowly absorbed. Abdominal pain, vomiting and diarrhea may be the only symptoms. However, if elimination is blocked by bowel blockage or other reasons, CNS depression, lack of reflexes, hypocalcemia (deficiency of calcium in the blood) may occur.
Skin Contact: No adverse effects expected but may cause minor skin irritation.
Eye Contact: No adverse effects expected but dust may cause mechanical irritation.
Incompatibilities: Furan-2-peroxycarboxylic acid. Strong oxidizing agents will release chlorine.

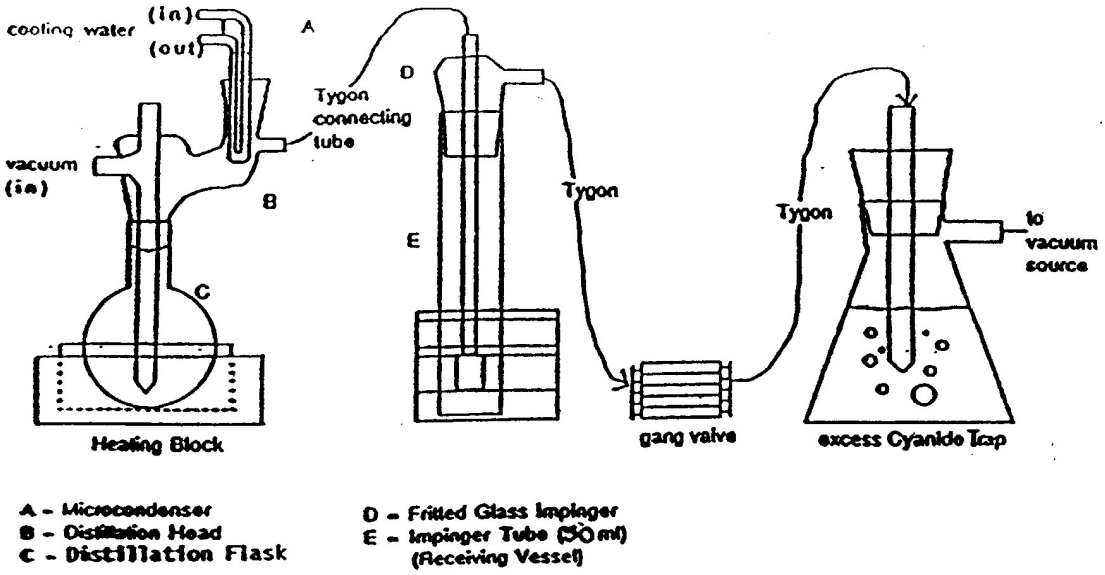

Figure 1

CYANIDE DISTILLATION APPARATUS

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Columbia Analytical Services SOP

C-1

STANDARD OPERATING PROCEDURE

for

SAMPLE DISPOSAL

SOP No.: SMO-SDIS

Revision: 7

March 26, 2008

Approved By:

Supervisor	3/26/08 Date
QA Manager	3-26-08 Date
Laboratory Manager	3/26/08 Date

COLUMBIA ANALYTICAL SERVICES, INC.
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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

SAMPLE DISPOSAL

1. PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe procedures for the disposal of unused portions of samples. Based upon the results of the analyses performed by the laboratory and other knowledge supplied by the customer. A sample is categorized as non-hazardous or potentially hazardous and this SOP describes procedures for disposal of each type. This categorization assumes contaminates requested for analyses are the only hazardous substances contained in the sample.

2. APPLICABILITY

The procedures for disposal described in this SOP applies to all sample types: aqueous, soil, solid, sediment, tissue, solvents, etc. The procedures for disposal described in this SOP also apply to all testing by-products.

3. DEFINITIONS

3.1. Sample: The material collected by a Customer in a container and delivered to the Laboratory for chemical analysis.

3.2. Testing By-Product: The material produced in the course of a chemical analysis, such as aqueous waste containing potentially hazardous substances.

3.3. Service Request: The document created by the laboratory at the time a sample is received at the laboratory and logged into the Laboratory Information Management System (LIMS). This document contains the client information, the sample identification and the instructions for the chemical analysis of the sample.

3.4. Custody record - External or internal COC document that clearly defines and documents who is/was in possession of a sample at a given time.

3.5. Login - The SMO process of accepting the sample, inspecting, and entering the sample information into the laboratory LIMS system.

3.6. Sample custodian – The sample management office (SMO) is considered to be the sample custodian for any period of time from sample receipt to sample disposal that the sample is not internally signed into custody by analytical personnel.

3.7. Sample tracking - Identifying or reproducing the path of sample custody throughout the laboratory.

4. RESPONSIBILITIES

4.1. It is the responsibility of the person or persons designated to categorize the samples (non-hazardous, potentially hazardous, etc.) that the determination is based upon the best available information and that the decision is based upon that information. It is also imperative that the assessment is done correctly versus established criteria.

4.2. It is the responsibility of the Sample Management Technician and designated Sample Custodian doing the disposal to follow the procedures described in this SOP so that the laboratory waste is properly, safely and legally disposed of. The Sample Management Technician and designated Sample Custodian have the responsibility of disposing of samples in the appropriate manner, based primarily on the hazardous criteria provided on the Service Request. This person will contact the assigned Project Chemist or the Safety Officer to resolve any uncertainties with regard to disposal. When in doubt as to the hazardous nature of a sample, the sample will be disposed of as hazardous.

5. PROCEDURES

5.1. Determination of sample composition

5.1.1. Service Request and Sample Receipt

A Service Request is created for each set of samples received at CAS. This Service Request describes the sample information, the work to be conducted and other pertinent information, including any special handling or hazardous disclosure information provided to us by the Customer. This Service Request also provides information to be filled out by a designated person or persons with respect to the hazardous nature of the samples.

5.1.2. Review of Analytical Results and Sample Categorization

5.1.2.1. Upon completion of chemical analyses by the laboratory, a certified analytical report is prepared for the customer that summarizes the analytical results obtained by the laboratory. This certified analytical report is typed, reviewed by the chemist and signed by the Project Chemist. After signing, the report is ready for distribution and the Service Request file containing all pertinent information concerning the analysis (including raw data) is ready for archiving.

5.1.2.2. At this time, the designated person or persons will compare the sample results in the certified analytical report (along with all potential hazardous materials information supplied by the Customer) versus the list of analytes in the Potentially Hazardous Materials Criteria (see Attachment I).

5.1.2.3. The designated person will indicate on the "Shipping Log" all of the samples that contained any known hazards above the listed limits as well as the appropriate waste stream for disposal (i.e. RCRA or PCB).

5.1.2.4. The sample custodian designates in the LIMS system all samples that are deemed hazardous from the "Shipping Log". The samples are labeled in the LIMS system as PCB, RCRA, Caustic, Corrosive, Halogenated, Non-Halogenated, Heat Sterilization waste.

5.2. Sample disposal

5.2.1. Pre-Disposal Holding

5.2.1.1. The sample management technician prints out a pre-disposal report from the LIMS system. From that list, all of the indicated service requests are printed. The technician determines the need for special sample archiving requirements by reviewing the information on the service requests and in the "special archiving" book.

5.2.1.2. The samples are pulled from the refrigerators and scanned into the LIMS system to assigned pre-disposal holding shelves. Aqueous samples are held for 31 days after the report has been mailed to the customer, and soils, sludge's, sediments, and solid samples are held for 60 days (unless special archiving arrangements have been made). Pre-disposal holding shelves are labeled with the report date and the date for disposal.

5.2.2. Sample Disposal

5.2.2.1. When samples are ready for disposal, the sample custodian removes the bottles from the pre-disposal shelves and places them on transport carts. Each sample container is scanned into the LIMS system as "properly disposed". The LIMS system indicates to the sample custodian the hazardous nature of the sample scanned. The waste stream is written with a sharpie on the cap or label of the bottle (i.e. PCB=P, RCRA=R, Caustic=C, Corrosive=Corr, Halogenated=Halo, Non-Halogenated=Non-Halo, Heat Sterilization =Auto, etc.). The sample is segregated in the appropriate satellite storage area. A maximum of 50 gallons can be accumulated in the satellite storage area before disposal into drums.

5.2.2.2. The completed Service Request Form, describing the hazardous nature of the samples is filed in the Sample Disposal notebooks.

5.2.3. Disposal of Non-Hazardous Samples

5.2.3.1. All non-hazardous, unpreserved, aqueous samples are flushed down the drain with large quantities of water. All preserved aqueous samples are neutralized via 25% NaOH or 10% HCL and then discharged down the drain with large quantities of water. A neutralization log is kept of all neutralized batches discharged to the sewer system.

5.2.3.2. Soil, sludge, sediment and solid samples that do not contain any known potentially hazardous chemicals can be discarded into the garbage bin for standard waste disposal. Alternatively, if requested by the Customer, the samples can be returned.

5.2.3.3. Disposal of Foul Condensate Samples

5.2.3.3.1. When SMO receives a cooler containing samples of Foul Condensate, the sample vials will be marked with a green sharpie across the bar code label (generated by the laboratory). Other sample ID's to include would be S.S. Feed, Stripped Condensate, turpentine.

5.2.3.3.2. At the time of sample disposal, these samples will be separated into a waste stream designated for treatment of samples containing high concentrations organosulfides.

NOTE: The following steps will take place in a fume hood.

5.2.3.3.3. A dilute bleach solution will be prepared in a 2-3 gallon plastic bucket. The samples are opened and emptied into the bucket.

5.2.3.3.4. The waste water can be disposed of down the drain with running water. The empty sample containers are disposed of inside a plastic garbage bag and put in the garbage for standard waste disposal.

5.2.4. Disposal of Potentially Hazardous Samples

5.2.4.1. If chemical analysis or Customer disclosure information indicates that potentially hazardous materials are present in the samples, the materials are separated into separate categories according to the types of hazardous waste – RCRA and PCB.

5.2.4.2. If samples are regular RCRA hazardous waste, these samples will be placed in a 55 gallon hazardous waste drum marked for RCRA waste. Alternatively, these samples can be returned to the Customer if requested.

5.2.4.3. All samples with PCB written on the lid will be placed in a drum marked PCB Waste Only or returned to the customer if requested.

5.2.4.4. All hazardous waste samples, whether they are RCRA or PCB waste must be emptied into the appropriate waste barrel.

5.2.5. Disposal of Other Sample Types

5.2.5.1. If a sample does not fall within the aqueous, soil/sludge or other categories described above, the Sample Management Technician will consult the assigned Project Chemist or the Safety Officer on proper method of disposal. These samples are to be disposed of following general waste disposal criteria and may be distinct from the sample disposal criteria described in this SOP.

5.2.5.2. All oil samples that do not contain PCBs or whose concentration is below the regulated level are to be placed into the RCRA waste drum.

5.2.5.3. Solvent samples are to be segregated as chlorinated and non-chlorinated and discarded into the appropriate 55-gallon waste drum marked chlorinated or non-chlorinated solvent waste. The waste solvent drums are then disposed of via an approved waste handling company.

5.2.5.4. Aqueous process chemical samples that are non-hazardous may be discarded down the drain in accordance with section 5.2.3.1. These chemicals may include soaps, defoamers, and other similar materials.

5.2.5.5. Dry chemical samples, such as sodium benzoate and other dry chemicals are to be retained as samples and/or returned to the Customer. These retained samples will then be "lab-packed" for disposal at a hazardous waste landfill.

5.2.6. Sample Container Disposal

5.2.6.1. Sample containers are not to have any identification on them that links or ties the container to CAS. Thus, containers provided to clients do not list the laboratory name. Clients should be encouraged not to list CAS or their name on the label.

5.2.6.2. For non-hazardous samples, the containers are dumped in the normal laboratory garbage containers. If the container does have laboratory identification on the label, either the container itself should be destroyed, or the label obliterated.

5.2.6.3. For hazardous waste samples, glass containers are included in the sample waste handling. Once emptied, plastic containers are dumped in the normal laboratory garbage containers. If the container does have laboratory identification on the label, either the container itself should be destroyed, or the label obliterated.

6. RECORDKEEPING

6.1. An internal chain of custody record of all sample disposal is recorded in the laboratory LIMS system.

Disposal of Non-Hazardous, Acidic or Caustic Water Samples

Waste Stream: Non-hazardous water sample disposal to sewer.

Date	Operator	Volume Disposed (L)	Neutralization Treatment Required	Final pH
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	
			25% NaOH 10% HCL None	

Attachment I

Hazardous Waste Criteria from CAS Environmental Health and Safety Manual

7.3 *Potentially Hazardous Materials Criteria*

Units:	WATER	1.0 ppb (ug/L) = 0.001 ppm (mg/L) = 1000 ppt (ng/L)
	SOIL	1 ppm (mg/Kg) = 1000 ppb (ug/Kg)
General Chemistry		
	pH	<2.5 = Acid waste
		>10 = Caustic waste
	Flashpoint	<140°F = Flammable
	Cyanide	Hazardous at any level = RCRA
	Reactive Sulfide	Hazardous at any level
	Formaldehyde	>100,000 ppb (ug/L) or 100 ppm (mg/L) = RCRA Water Drum
	TOX, EOX, AOX	>100 ppm (mg/Kg) = RCRA Soil Drum
		>100,000 ppb (ug/L) or 100 ppm (mg/L) = RCRA Water Drum
	Hexavalent Chromium	Hazardous at any level

Metals

	SOIL Mg/Kg (ppm)	WATER ug/L (ppb)	WATER mg/L (ppm)	TCLP mg/L (ppm)
Arsenic	>100	> 5000	> 5	> 5
Barium	>2000	> 100000	> 100	> 100
Cadmium	>20	> 1000	> 1	> 1
Chromium	>100	> 5000	> 5	> 5
Lead	>100	> 5000	> 5	> 5
Mercury	>4	> 200	> 0.2	> 0.2
Selenium	>20	> 1000	> 1	> 1
Silver	>100	> 5000	> 5	> 5
Hexavalent Chromium	-	Hazardous at any level		-

Pesticides -

	SOIL Mg/Kg (ppm)	WATER ug/L (ppb)	WATER mg/L (ppm)	TCLP mg/L (ppm)
Chlordane		> 30	> 0.03	> 0.03
Endrin	> 0.4	> 20	> 0.02	> 0.02
Heptachlor		> 8	> 0.008	> 0.008
Heptachlor Epoxide		> 8	> 0.008	> 0.008
Methoxychlor				> 10
Lindane	> 0.8	> 400	> 0.4	> 0.4
Toxaphene	> 10	> 500	> 0.5	> 0.5

Herbicides

	SOIL Mg/Kg (ppm)	WATER ug/L (ppb)	WATER mg/L (ppm)	TCLP mg/L (ppm)
2,4 -D	> 200	> 10000	> 10	> 10
2,4,5-TP (Silvex)	> 20	> 1000	> 1.0	> 1.0

Semivolatile - 8270,

	SOIL Mg/Kg (ppm)	WATER ug/L (ppb)	WATER mg/L (ppm)	TCLP mg/L (ppm)
Hexachloroethane	> 60	> 3000	> 3	> 3
Nitrobenzene	> 40	> 2000	> 2	> 2
Hexachlorobutadiene	> 10	> 500	> 0.5	> 0.5
2,4 -Trichlorophenol	> 2.6	> 130	> 0.13	> 0.13
Hexachlorobenzene	> 2.6	> 130	> 0.13	> 0.13
2,4,6 -Trichlorophenol	> 40	> 200	> 2	> 2
2,4,5 -Triclorophenol	> 8000	> 400000	> 400	> 400
Pentachlorophenol	> 2000	> 100000	> 100	> 100
Pyridine	> 100	> 5000	> 5	> 5
Cresols (o,m,p, total)	> 4000	>200000	>200	>200

Total Halogens >100 ppm (mg/Kg) = RCRA Soil Drum

>100,000 ppb (ug/L) or 100 ppm (mg/L) = RCRA Water Drum

Total PAH >10,000,000 ppb (ug/L) or 10,000 ppm (mg/L) = RCRA Water Drum

PCBs

>2ppm < 50 ppm (mg/Kg or mg/L) or < 50000 ppb (ug/L) = RCRA PCB Drum

> 50 ppm (mg/Kg or mg/L) or > 50000 ppb (ug/L) = TOSCA PCB Drum

Dioxins

Hazardous at any concentration = RCRA PCB Drum

PHC

	SOIL Mg/Kg (ppm)	WATER ug/L (ppb)	WATER mg/L (ppm)	TCLP mg/L (ppm)
Diesel	> 100	> 100000	> 100	
Gasoline	> 100			
Methanol	> 100			
Oil & Grease	> 100			

Explosives

SOIL mg/Kg (ppm)	WATER ug/L (ppb)	WATER mg/L (ppm)	TCLP mg/L (ppm)
> 100	> 100000	> 100	

VOA

	SOIL mg/Kg (ppm)	WATER ug/L (ppb)	WATER mg/L (ppm)	TCLP mg/L (ppm)
Benzene	> 10	> 500	> 0.5	> 0.5
Carbon Tetrachloride	> 10	> 500	> 0.5	> 0.5
Chlorobenzene	> 2000	> 100000	> 100	> 100
Chlordane	> 0.6	> 30	>0.03	>0.03
Chloroform	> 120	> 6000	> 6.0	> 6.0
1,4 - Dichlorobenzene	> 150	> 7500	> 7.5	> 7.5
1,2 - Dichloroethane	> 10	> 500	> 0.5	> 0.5
1,1 - Dichloroethylene	> 14	> 700	> 0.7	> 0.7
2,4 - Dinitrotoluene	> 2.6	> 130	> 0.13	> 0.13
Hexachlorobenzene	> 2.6	> 130	>0.13	>0.13
Methyl Ethyl Ketone	> 4000	> 200000	> 200	> 200
Nitrobenzene	> 400	> 2000	> 2.0	> 2.0
Tetrachloroethylene	> 14	> 700	> 0.7	> 0.7
Trichloroethylene	> 10	> 500	> 0.5	> 0.5
Vinyl Chloride	> 4	> 200	> 0.2	> 0.2
BTEX	> 100	> 100000	> 100	>100
Benzene	> 10	> 20000	> 20	
Toluene	> 20	> 20000	> 20	
Ethylbenzene	> 20	> 20000	> 20	
Xylene	> 20	> 20000	> 20	
MTBE	> 100	> 100000	> 100	>100

- Based on Washington State Criteria

Table 7-2—Hazardous Materials Criteria

C-2

STANDARD OPERATING PROCEDURE

for

SAMPLE TRACKING AND INTERNAL CHAIN-OF-CUSTODY

SOP No.: SMO-SCOC

Revision: 11

March 21, 2008

Approved By: _____ Supervisor — 3/21/08 Date

_____ QA Manager — 3-21-08 Date

_____ Laboratory Manager — 3/21/08 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

SAMPLE TRACKING AND INTERNAL CHAIN-OF-CUSTODY

1. PURPOSE

The purpose of this procedure is to maintain and document internal chain-of-custody (COC) of samples. Custody documentation must be maintained through all phases of analysis - from sample receipt and initial storage to analysis and disposal.

2. APPLICABILITY

The procedure is applicable to all field samples, subsamples, and sample extracts and sample digestates as described in the procedure section.

3. DEFINITIONS

3.1. Custody - The guardianship or safe keeping of a sample. A sample is considered to be in a person's custody if it is physically in their possession, or it is in their view after being in their possession, or it was in their possession and then locked up or sealed to prevent tampering, or it is in a secure area.

3.2. Chain of Custody - The documented order or sequence of the persons having custody of a sample. Chain of custody requires that the sample be in custody of some known person from the time it is collected until it is disposed.

3.3. Custody record - External or internal COC document that clearly defines and documents who is/was in possession of a sample at a given time.

3.4. Login - The SMO process of accepting the sample, inspecting, and entering the sample information into the laboratory LIMS system.

3.5. Sample custodian – The sample management office (SMO) is considered to be the sample custodian for any period of time from sample receipt to sample disposal that the sample is not internally signed into custody by analytical personnel.

3.6. Sample tracking - Identifying or reproducing the path of sample custody throughout the laboratory.

4. DISCUSSION

It is important to quality analyses to have the sample custody verifiable with meaningful documentation. Procedures that ensure proper sample handling and accurate and complete documentation are a must in order to provide our customers with data that is legally defensible.

Legal defensibility of our analytical data begins with an unbroken chain of custody from the time of sample collection until sample disposal. The establishment of chain of custody procedures is of paramount importance, since the results of our testing or analysis might eventually be held as evidence in a trial or hearing.

5. RESPONSIBILITIES

It is the responsibility of sample custodians and laboratory staff to make sure that the procedures described in this SOP are followed and that chain of custody records are complete and accurate. Every appropriate laboratory staff member will be required to familiarize themselves with the procedures listed in this SOP. Once the SOP has been read, appropriate supervisory personnel and section managers must ensure that personnel will comply with the procedures listed herein.

6. PROCEDURES

6.1. Field sample custody

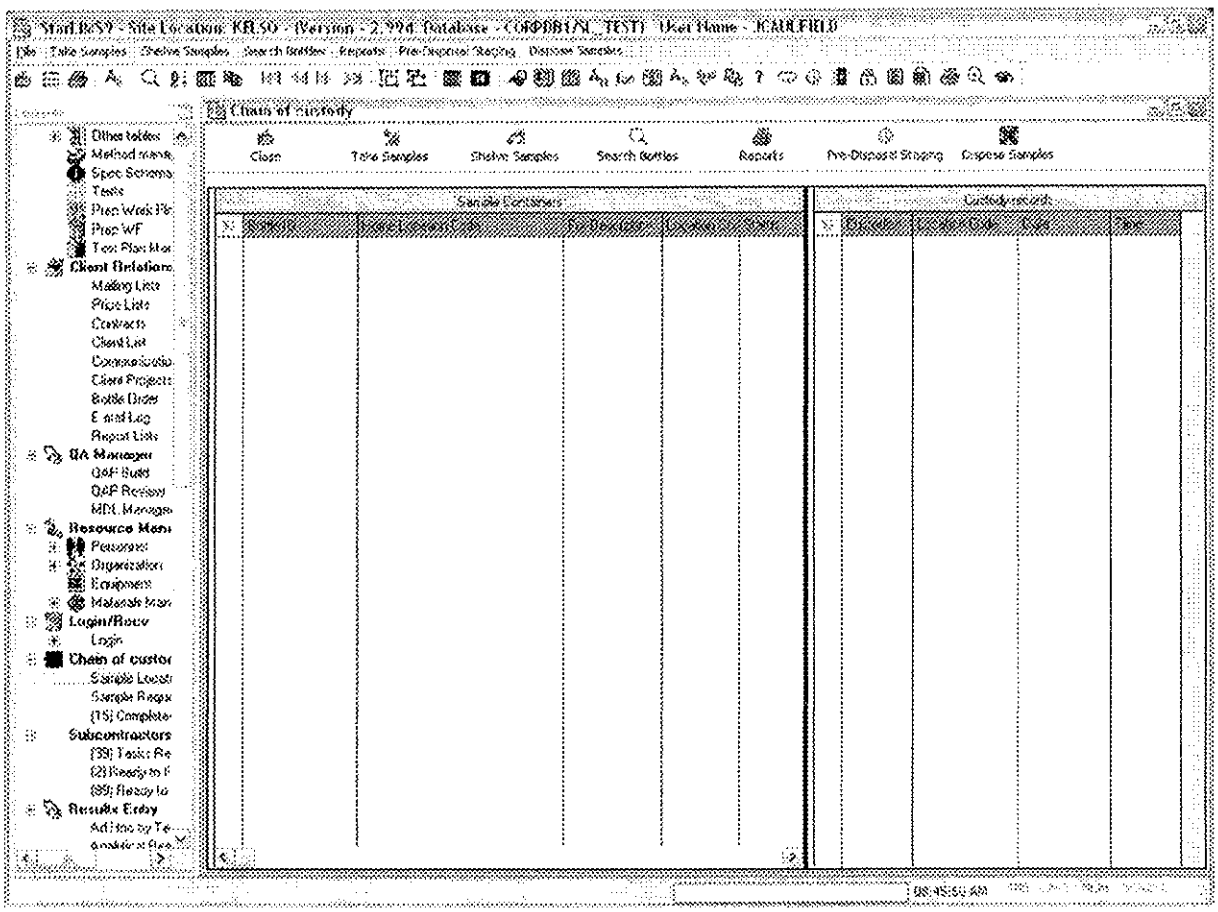
6.1.1. A bar-coding system is used to identify, label, and track field samples throughout the laboratory. The bar-coding system uses Access© database for sample information. The sample tracking process in the laboratory begins with receipt of samples into the laboratory facility. Sample receipt, login, identification, labeling, and storage procedures are all described in the SMO-GEN Standard Operating Procedure. Sample receiving personnel will enter the sample information (Service Request number, unique container ID) for each project into the sample database. Following login, the samples are placed in the appropriate refrigerator(s).

6.1.2. Access to all samples is controlled. Samples are stored in a secure, keypad-locked room or in separate locked storage areas. While in these secured and locked storage areas, the samples are considered to be in the custody of the sample management office (sample custodian). Removal of any sample from the SMO sample storage areas represents a physical change of custody and therefore must be documented via a chain of custody record. It is at this point that internal tracking of the samples is initiated.

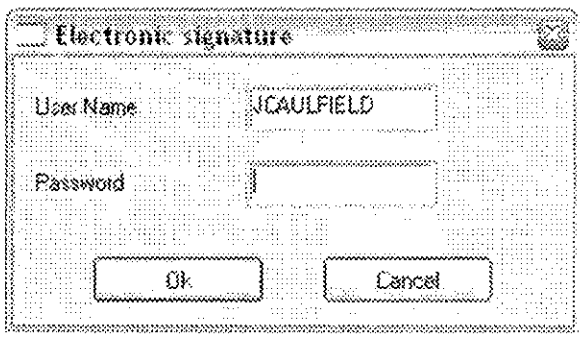
Note: All solid samples pulled from a freezer must be placed in a Ziploc storage bag to prevent loss of sample in the event that the jar cracks during thawing.

6.1.3. Each employee with access to samples is assigned a unique barcode ID which represents their electronic signature. The steps to be taken to check out samples (into the analyst's custody) are outlined below:

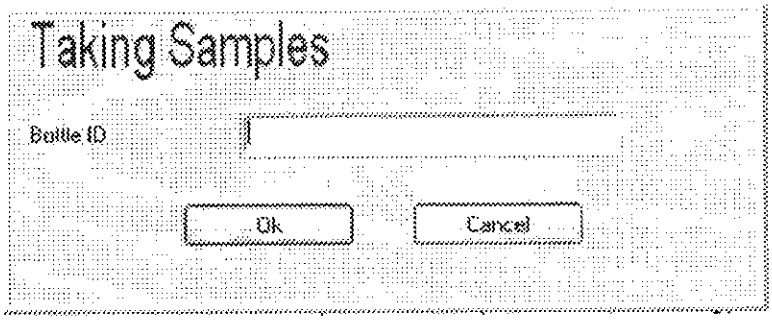
6.1.3.1. The main menu display on the internal custody workstation appears as follows:



6.1.3.2. Choose Take Samples to check samples out of storage. The following Signature window will appear:



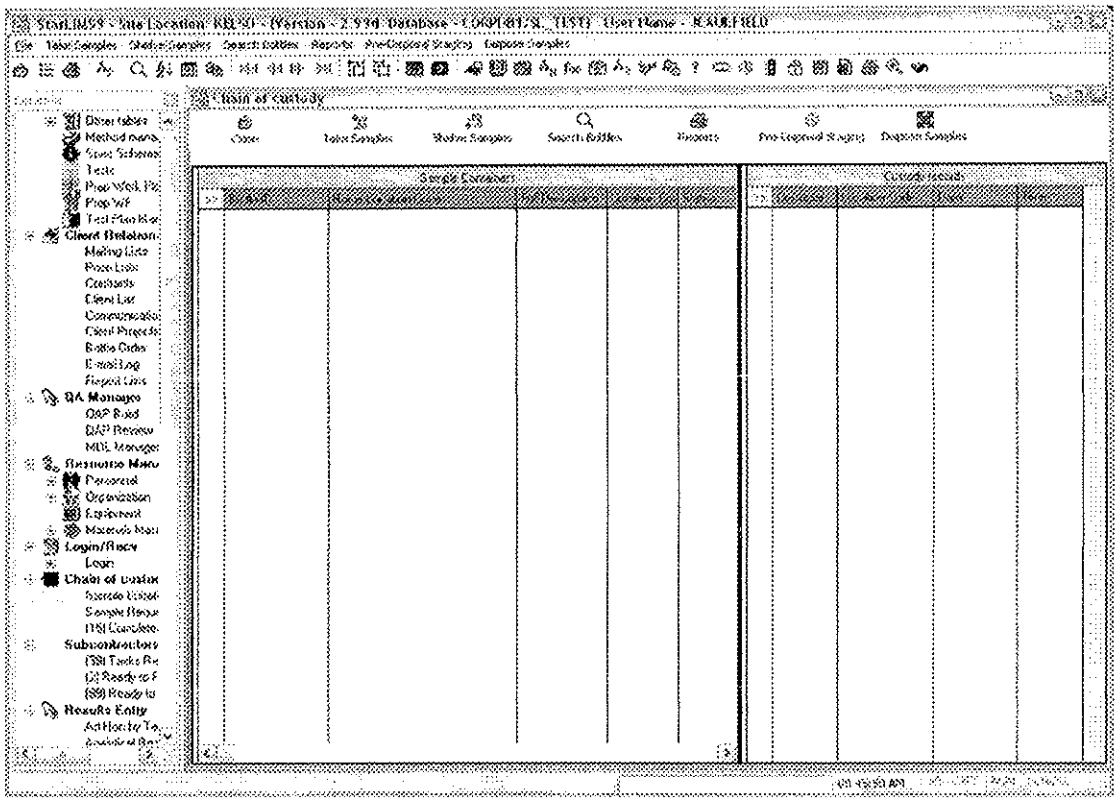
6.1.3.3. Select name from employee list. The Taking Samples screen will appear next.



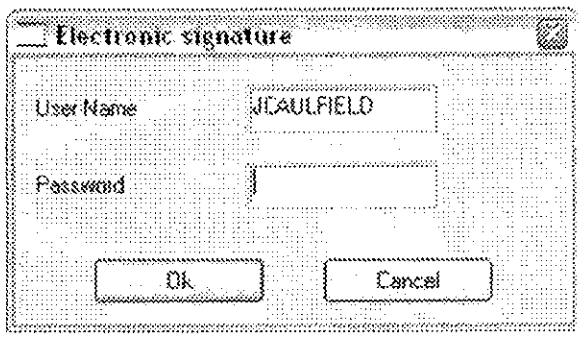
6.1.3.4. Using the barcode reader, read the barcode for each sample to be removed and taken into custody. The sample list will appear in the window displayed. It is recommended that a brief comparison of the displayed list and the actual container IDs be made.

6.1.4. Returning samples

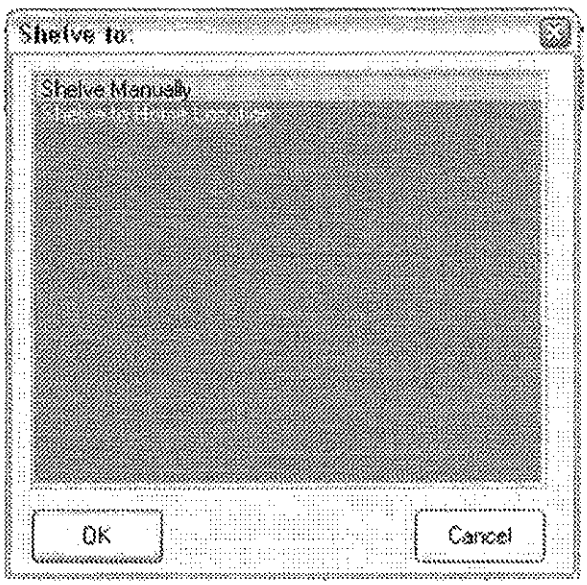
6.1.4.1. When the analyst is finished working with the samples, the containers are checked back in to complete the custody record and document the return to custodian custody. On the main menu display, choose Shelve Samples.



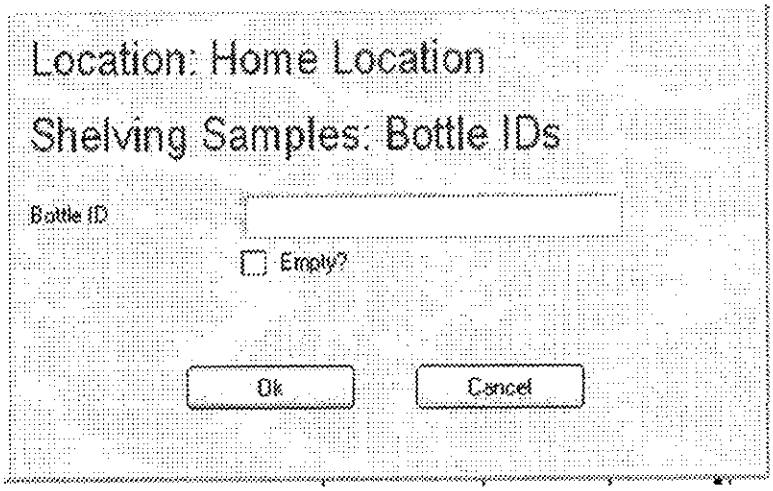
6.1.4.2. The Employee Name window will appear:



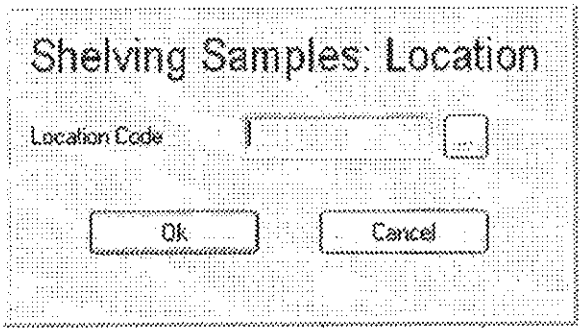
6.1.4.3. Select name from employee list. The Shelve To screen will appear next. Choose whether to shelve to home location or manually.

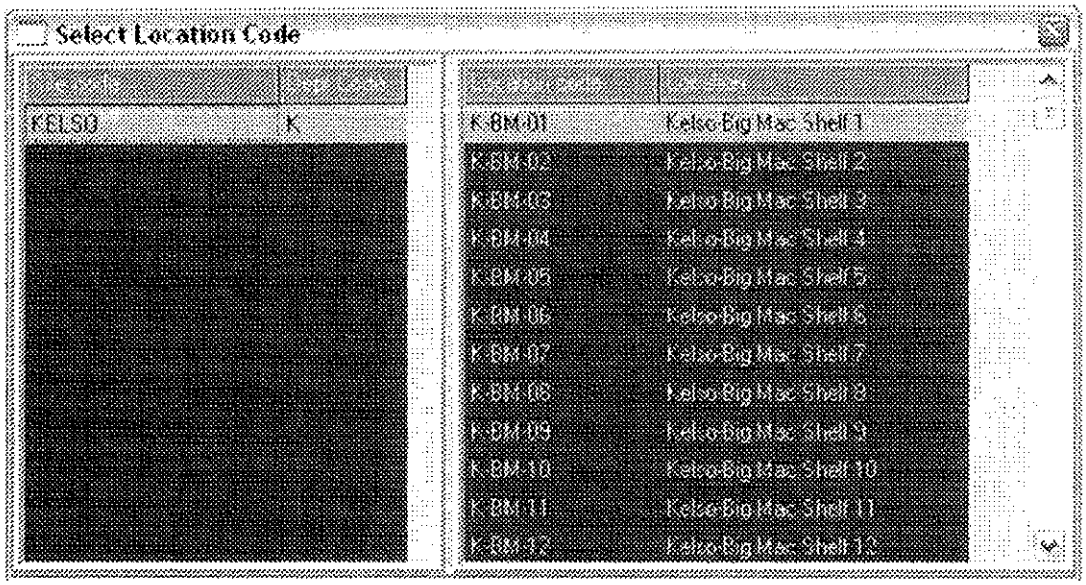


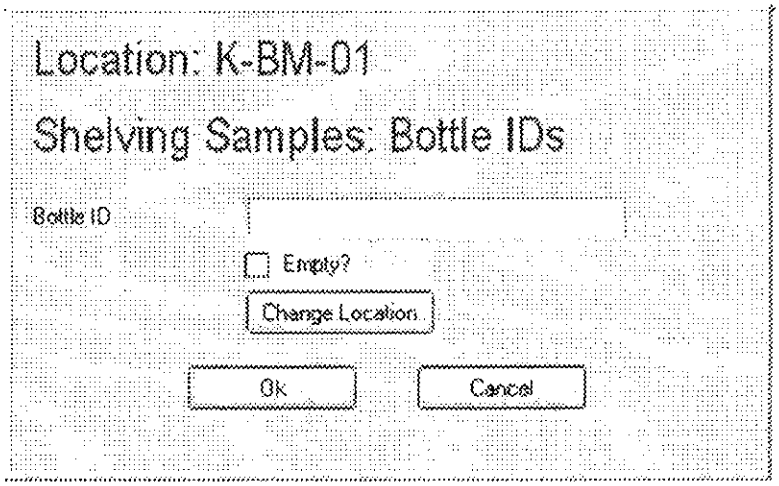
6.1.4.4. If Home Location is chosen, use the following screen and scan the samples. For each container, check the Empty checkbox if the container is empty.



6.1.4.5. If Manual is chosen, use the following screens to choose the sample location. Scan the samples. For each container, check the Empty checkbox if the container is empty.



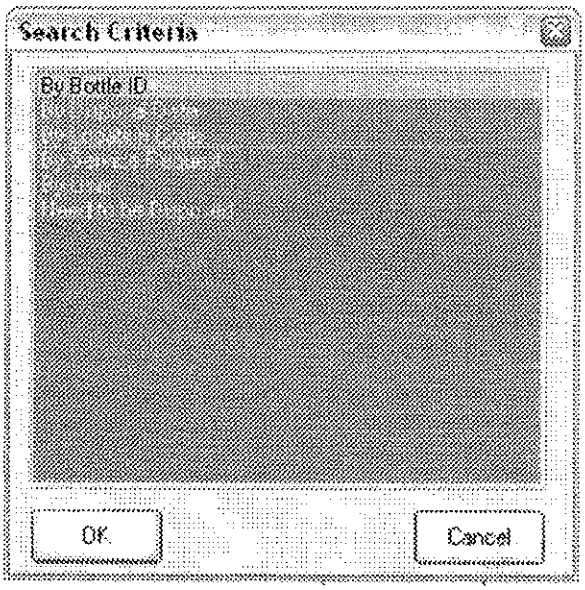




6.2. Sample tracking

6.2.1. Sample custody can be tracked via the Search function. Using this function, an analyst looking for a container that is in another analyst's custody can locate the container. The function is also used to document all custody events and provide internal chain of custody history. At the main menu window, choose Search Bottles. The Search window will appear.

6.2.2. The search can be done several ways by choosing one of the criteria in the Search Criteria window. The window will appear with all custody information.

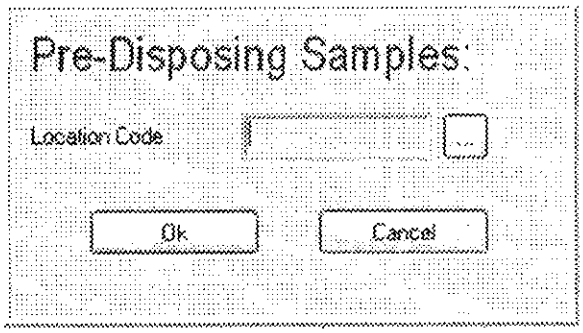


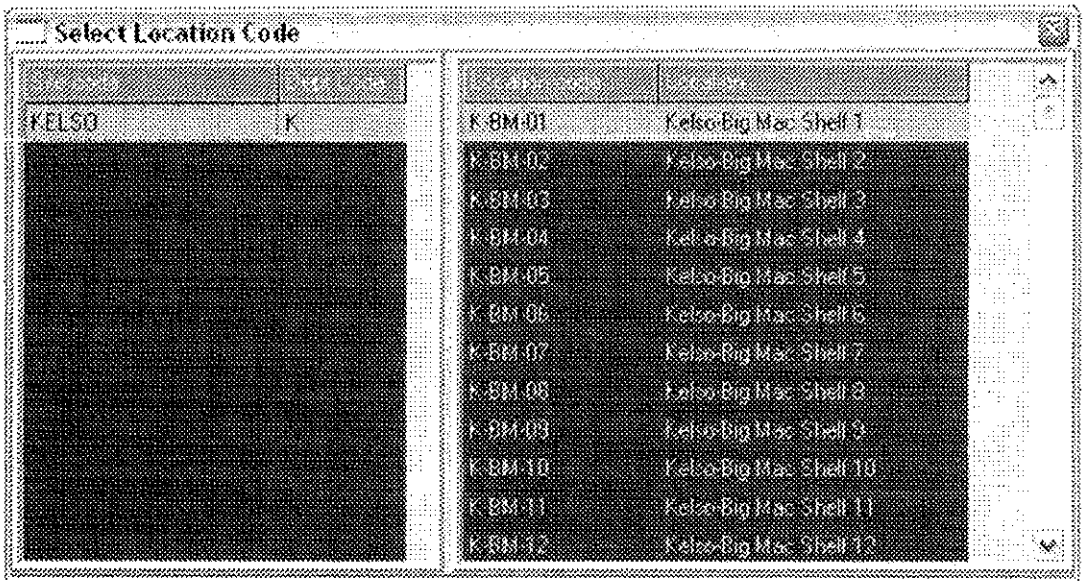
6.3. Custody of extracts and digestates

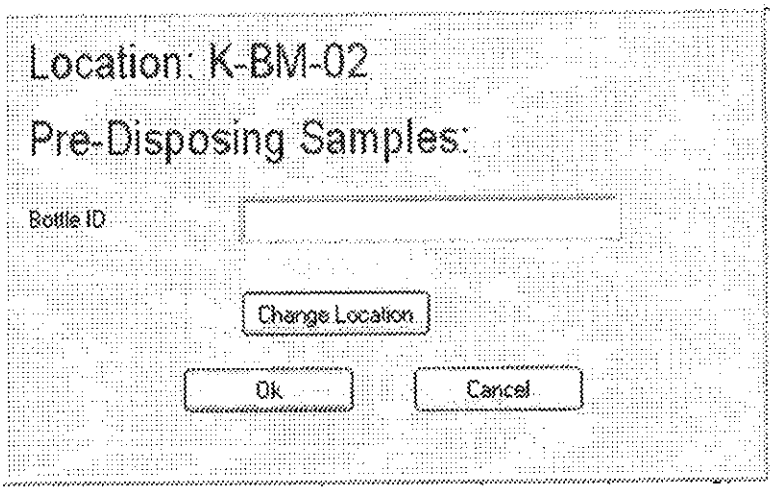
6.3.1. In labs where separate analysts prepare samples (extraction or digestion) for instrument analysis, the transfer of custody of completed extracts or digestates is documented. The instrument lab analyst accepts custody of the sample extract or digestate by signing on the benchsheet, or similar custody record, that they have accepted custody of those specific extracts or digestates listed. The date and time must accompany the signature.

6.3.2. For TCLP extracts, the finished extracts are labeled and logged into the LIMS system as a new container for the sample. The extracts are then stored as with other samples. Custody and tracking is then performed as for field samples as discussed in sections 6.1 and 6.2.

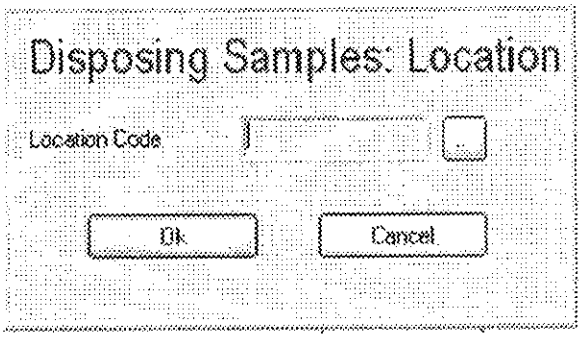
6.4. When the analytical report has been shipped to the client, the sample containers are removed from refrigeration and placed on pre-disposal holding shelves while still in the custody of SMO. The shelves are referenced in order to facilitate retrieval of a sample if necessary. Transfer is documented using the Pre-Disposal and Location Selection windows.

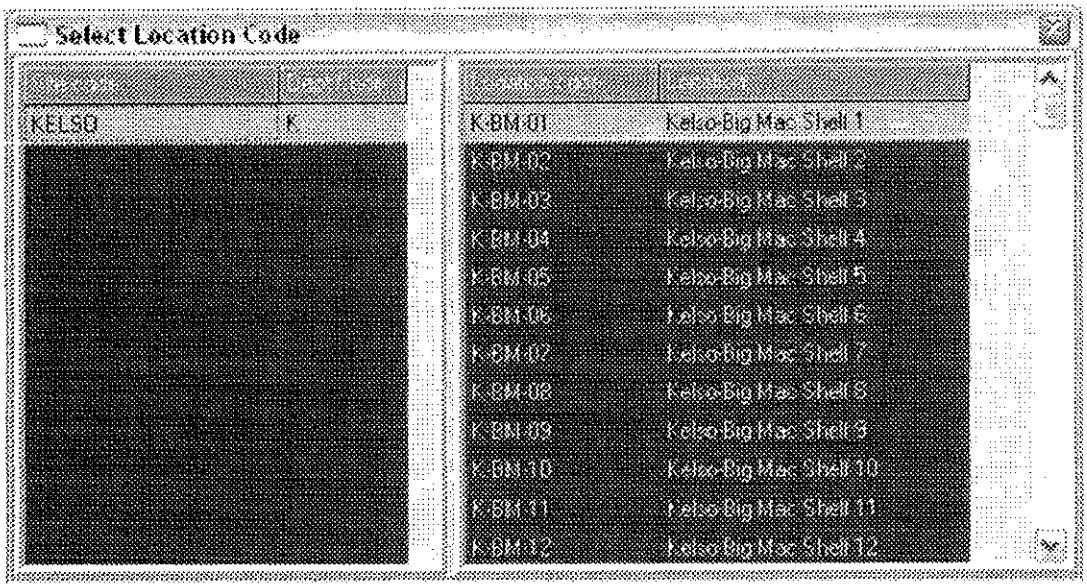


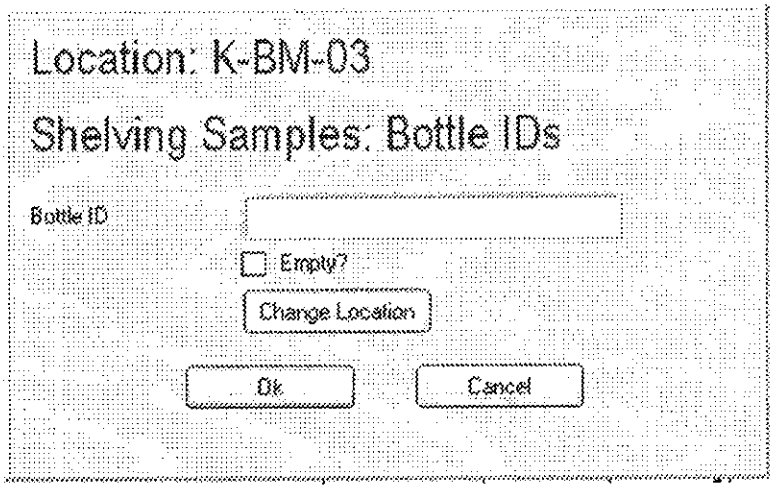




6.5. When sample disposal is performed, each container is scanned. Thus, the endpoint of CAS custody is documented by the final scan and documented disposal date (refer to the SOP for Sample Disposal).



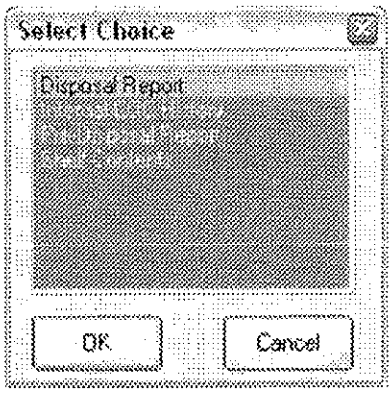




7. RECORDS AND REPORTS

7.1. Each service request will contain one or more references to the location of all of the associated sample containers. The record of hazardous/non-hazardous sample determination is kept in the original service request file.

7.2. After analyses are completed for the entire Service Request and the data is reported, the entire sample custody record is printed and placed in the original SR file. This, and other available reports, is found in the report selection window.



7.3. Human resources and QA records are used to associate the analyst's barcode signature to their actual handwritten signature. The database of internal custody records will be maintained for 5 years. Normal CAS procedures for electronic backup are used.

C-3

STANDARD OPERATING PROCEDURE

SAMPLE RECEIVING

SMO-GEN

Revision 24

March 5, 2008

Approved By:

Supervisor	3/5/08 Date
QA Manager	3.5.08 Date
Laboratory Manager	3/8/08 Date

COLUMBIA ANALYTICAL SERVICES, INC.
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Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

SAMPLE RECEIVING

1. PURPOSE

This procedure is used to facilitate the correct and expedient receipt, login, and storage of all samples received at CAS.

2. SCOPE AND APPLICATION

This procedure applies to all samples received at CAS. This procedure applies to all phases of sample handling once received, including checking and documentation of sample condition, documenting sample custody, sample login into the Laboratory Information Management System (LIMS), storage, shipping, and disposal

3. RESPONSIBILITIES

3.1 It is the responsibility of all lab staff who handle samples to adhere to this procedure and to complete all documentation required. It is the responsibility of the department supervisor/manager to ensure that SMO staff are trained on this procedure and that training documentation is available. It is also the responsibility of the SMO supervisor to ensure that a secondary review of all sample login documentation is performed.

3.2 It is the responsibility of all CAS employees to ensure the confidentiality of client information. All information, such as customer name and project information, data, and reports is treated as confidential and is to be kept out of sight of other customers. No information may be given to third parties without the consent of the client.

4. DISCUSSION

4.1 The quality of analytical results is highly dependent upon the quality of the procedures used to collect and handle, and store samples. Therefore, sample management is a critical component of the laboratory operation. This procedure ensures that samples are properly received and logged into the Laboratory Information Management System (LIMS), and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received. Complete documentation of all sample storage is maintained in order to preserve the integrity of the samples and to establish a record of sample handling. All potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues.

4.2 During the log-in process, each sample is given a unique laboratory code and a service request form is generated. The service request contains client information, sample

descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information.

5. PROCEDURE

5.1 Sample receipt, unpacking, and inspection

5.1.1 Upon receipt, sample shipping containers are examined for presence and condition of custody seals, locks, shipping bills, etc. Before processing, all shipping containers (coolers) are initially opened in the hood. The cooler is quickly inspected for any existing hazards such as broken bottles and a thermometer is inserted. The CAS Cooler Receipt and Preservation Form (CRF – see Appendix) is used for the next steps. CAS follows EPA guidelines for preservation and holding time as outlined in the CAS Quality Assurance Manual Table 8-1.

NOTE: If the cooler or any sample exhibits a strong odor, or there is any indication of highly contaminated samples, immediately transfer the cooler/samples to the hood if not already opened there. Ensure that appropriate protective equipment is being used (lab coat, gloves, eye protection, respirator if necessary, etc.). Continue to unpack the cooler. Leave the samples under the hood with the hood running. Notify the Project Chemist of the sample condition. For waste or product samples, store the samples in the hood in SMO. For other samples (water, soil/solid, etc) store the samples in the walk-in cooler(s). If any of the suspect or highly contaminated samples are designated for VOA analysis, DO NOT store with the other VOA samples in the VOA sample refrigerator. Discuss storage options with the Project Chemist and document the storage area/conditions.

5.1.2 After opening shipping containers, Chain of Custody (COC) forms and any other documents are located and removed. The temperature of the cooler is measured following the guidelines in section 5.4 of this SOP. The acceptance criteria for properly refrigerated samples in a cooler is $4 \pm 2°C$. If a cooler exhibits a temperature outside of the acceptance range, or exhibits any other anomalies, the CRF is attached to the Service Request and submitted for review to the appropriate project manager. Samples or coolers which are received outside of the temperature range are noted on the cooler receipt form.

NOTE: EPA drinking water requirements for microbiology samples (surface water treatment rule and coliphage samples) require that samples be at 10°C or less. If the temperature is $> 10°C$, the sample results must be flagged by recording the temperature in the "'Remarks" section of the Bacteriological Analysis Form, or by indicate the temperature and flagging requirement on the CRF if the results will be reported on standard forms.

NOTE: Some states or projects may require specific cooling requirements for shipping coolers and special cooler temperature assessment procedures. For example, Wisconsin DNR does not allow for use of blue ice. For WDNR, it should be noted if regular ice was present in the cooler.

5.1.3 Samples received with short or expired holding times will be issued via a "Temporary Service Request", prior to the final Project Chemist review. This enables the SMO staff to quickly notify lab staff of the samples. For samples that require wastewater microbiology testing, the analysis is to begin as soon as possible after receipt at the laboratory. Holding times are assigned according to the sample type, test method, and as prescribed in Standard Methods. Microbiology samples received past the holding time do not meet sample acceptance requirements and a notation is made on the Bacteriological Analysis Form and/or Cooler Receipt and Preservation Form. Decisions regarding the disposition of the samples that do not meet sample acceptance criteria are to be documented on the Cooler Receipt and Preservation Form by the Project Chemist.

5.1.4 Rush requests and samples with short holding times are always given priority for initial processing. Sample containers are removed and organized according to chain of custody identification and analysis. Any containers that are received in broken condition or leaking are documented on the Cooler receipt Form (CRF) and on the chain of custody. The appropriate Project Chemist is also notified. For EPA samples which exhibit discrepancies or are damaged upon receipt, the EPA Project Chemist is responsible to notify EPA SMO for direction.

Note: Samples received for NCASI Method WP-99.02 must be kept refrigerated or iced in accordance with section 6.3 of the method SOP. Once the samples are identified and verified against the COC, the samples must be placed back in an iced cooler or a refrigerator until login is complete and samples can be relocated to their assigned storage location. **DO NOT DELIVER SAMPLES TO THE LABORATORY**.

5.1.5 For water VOA samples (those received in 40mL VOA vials) and samples for method 1631 analysis the inspection includes checking the vials for headspace. Refer to section 5.1.8.4 for guidance.

5.1.6 Listed below are items which must be verified against the chain of custody information as it applies to samples and containers received:

- Sample identification
- Number of containers received

- Matrix
- Correct bottles for analysis requested.
- Correct/sufficient sample volume for analysis
- Correct preservatives for analysis. The CAS sample bottle preparation form is attached as a reference for proper preservatives.

At this point, a brief evaluation of the amount of sample provided for the requested analyses is made. Compare the sample containers and estimated amount of sample provided to the analyses requested on the COC. Using Table 1, determine if the amount of sample provided is at least as much as the minimum sample amount required. If not, notify the Project Chemist. The Project Chemist should provide further instructions for handling the sample login.

5.1.7 For inorganics analyses on water samples where preservation at a prescribed pH is required, check for the appropriate pH. Refer to Table 2 for those analyses requiring the pH check at the time of receipt. For example, water samples for metals analysis require a pH of <2 and water samples for cyanide analysis require a pH of >12. The pH check is documented on the CAS sample log-in form which, when completed, is kept with the SDG file. The pH is checked on bottles requiring certain organics analyses, including oil and grease, hydrocarbon, or VOA analyses by the lab department. All pH measurements made by SMO personnel must be performed by pouring a small aliquot of sample from the bottle into a plastic weigh boat. Narrow-range (increments of 0.2 units or less) pH test strips are used. The pH test strips are dipped into the liquid in the weigh boat in order to measure the pH of the sample. Under no circumstances is a pH test strip to be dipped directly into a sample container, due to the possibility of contamination. If further preservation of the sample is needed, the appropriate acid or base is added (up to a maximum of 2mL per liter). The lot number for the preservative added is recorded on the cooler receipt and preservation form.

Note: If acid is added to a metals sample at the time of login, a comment is added to the Service Request indicating to the analyst that the sample was preserved at login. This indicates to the analyst that the 16-hour minimum sample holding requirement listed in the laboratory SOPs applies.

5.1.8 For semivolatile organics analyses on water samples where preservation is necessary for samples with residual chlorine present, check for the presence of residual chlorine (see Table 2 for analyses required). Using a potassium iodide starch test strip, pour a small amount of sample into a weigh boat and dip the strip into the weigh boat. If positive, the test strip will turn purple. Test one bottle per sample and record the result. If the test for residual chlorine is positive, the sample is taken to the laboratory for addition of sodium thiosulfate solution (1mL of 10% to each applicable bottle) and documented. The lot number

for the preservative added is recorded on the cooler receipt and preservation form.

5.1.9 Discrepancies and Broken Samples

5.1.9.1 Any discrepancies or concerns from sections 5.1.1 through 5.1.7 above are noted on the COC and/or Cooler Receipt Form and communicated to the Project Chemist. Any resolutions are recorded on the Chain of Custody form, a nonconformity form, or a phone log that is placed in the service request master file. If a Chain of Custody is not received, information is noted on a CAS Log-In Information sheet (see Appendix).

5.1.9.2 When sample identifications on bottles are different than what is on the Chain of Custody, the difference is noted on the Chain of Custody and CRF. The project chemist is informed. If the sampling time is not listed on the COC, transcribe the time from the bottle to the COC. Once it is logged in on the service request the way the client wants and discrepancies are are corrected and noted on the Chain of Custody, a red check is placed on the sample bottle. This informs the lab that the discrepancy has been noted. If there is a discrepancy between the service request identification and the sample bottle identification and no red check is on the sample bottle, the analysts must inform SMO immediately to verify they have the right sample and it can be documented as stated above.

5.1.9.3 In the event of broken samples, a note is entered on the Chain of Custody and/or the cooler receipt form accompanying the samples. Information pertaining to the sample is forwarded to the project chemist for follow up purposes. Cleanup procedures are as follows:

Liquids: Broken glass is handled carefully using disposable gloves and disposed of in the Glass Disposal Box. The liquid is disposed of in the SMO sink under a hood. Any packing material is disposed of appropriately.

Soils: The same documentation as liquids applies. Broken glass is disposed of in a Glass Disposal Box and the soil is disposed of into the hazardous soil bucket for disposal. This information is noted on the Chain of Custody and CRF.

5.1.9.4 For VOA samples in VOA vials, EPA Method 5030B states that the size of any bubble caused by degassing upon cooling should not exceed 5-6mm. The presence of air bubble(s) is noted on the CRF if the bubble is $\geq$ 5mm (approximately the size of a small pea). If a bubble this size is present, the cap and septum should be inspected for breakage or leakage.

The Project Chemist will be notified if no other vials for the sample can be used (all vials have bubbles). For method 1631 samples, inspect the sample for the presence of headspace. Document on the CRF if present.

5.1.10 For EPA CLP work, the following additional steps must be taken:

5.1.10.1Designated Sample Custodian

The SMO department must designate a primary sample custodian who is responsible for the log-in of EPA CLP samples. An alternate representative should also be designated; the EPA PC should be aware of the identity of these people and should be notified if there is a desire to transfer this authority to a different individual.

5.1.10.2Sample Log-In Sheet (DC-1)

This form is used to document receipt and inspection of samples and containers. A DC-1 form (see Appendix) is required for each cooler. It must be completed as the cooler is opened and samples are examined, as described in the applicable CLP SOW. In addition to examining the items found in section 5 of this SOP, the following additional items must be reviewed and/or noted on form DC-1:

- The presence/absence and condition of custody seals.
- The presence/absence of chain-of-custody forms, airbills or airbill stickers, traffic reports or packing lists, and sample tags.
- Custody seal numbers when present
- Airbill or airbill sticker numbers
- Sample tags listed/not listed on the COC records
- Date and Time of receipt
- EPA sample numbers
- Sample tag numbers

When the DC-1 form is completed, the sample custodian or designated alternate must provide a full signature sign-off on the report; initials are not acceptable.

5.1.10.3pH Verification - Check the pH as described in section 5.1.6. Any discrepancy from this must be reported to EPA SMO.

5.1.10.4Sample Traffic Reports

A traffic report is required for each Sample Delivery Group. Traffic reports and an SDG cover sheet must be sent to SMO within three days of receipt of the last sample in SDG, as described in the applicable CLP SOW.

5.1.10.5SDG Naming

For EPA CLP work, the following rules must be followed in forming Sample Delivery Groups (SDG). An SDG is defined by the following, whichever is more frequent:

- Each case of field samples, or
- Each 20 field samples in a case, or
- Each 14 calendar day period during which field samples in a case are received. The 14-day period begins with the receipt of the first sample.

The SDG number is the 6-digit EPA sample number of the first sample received in an SDG. When several samples are received at the same time, the lowest alpha, numeric number is used. CAS assigns a unique lab code to each sample. The lab codes are recorded on the Chain of Custody where it is applicable.

5.1.11 Samples received after normal business hours (8-5 Monday – Friday and 8-12 on Saturday) are stored in a cooler in an appropriate refrigerator and are fully processed the following day. Upon receipt, an After Hours Receipt Form is completed and the temperature of the cooler is recorded in accordance with section 5.4 of this SOP (Note: the temperature must be recorded from the temperature blank if one is present). If samples are received the same day they are collected they can be placed in appropriate cold storage without determining cooler temperatures (In this case, samples are considered acceptable if there is evidence that the chilling process has begun and they do not need to meet the criteria of being within 2° C of the required temperature). The location of storage is noted on the After Hours Receipt Form (i.e. Samson, Herk, etc.).

5.1.12 The chain of custody form will be signed when the log-in procedure is completed (on the next working day if the samples are received after hours).

5.1.13 Courier Service

CAS provides a Courier Service for our Clients from our Kelso Laboratory Southbound to the Portland Metropolitan area to pick up samples. When picking up samples from Clients, a Courier/After-Hours Sample Receipt Record is filled out and signed by our Courier and also by the Client/Sampler. Information such

as date & time of pick-up, the Company name, number of containers, project number and if custody seals were present or added are noted on this Receipt Form. The Courier retains a copy of the Courier/After-Hours Sample Receipt Record and this copy is placed in the Master File once the job is logged into the LIMS system in the laboratory. The original copy is given to the Client at the time the CAS Courier takes possession of the samples.

5.2 Sample Login

5.2.1 Each job received at the laboratory is logged into the Laboratory Information Management System (LIMS). Access to LIMS Login is limited to Sample Management Staff and Project Chemists. The LIMS will assign the next available Service Request number. The Service Request number is made up to identify the CAS lab, the year and then a numeric number (i.e. K0502250: K for the Kelso lab, 05 for the year and then 02250 would be the next number available in the system).

5.2.2 The client and project are identified in the LIMS based on the COC information. The Sample Delivery Group (SDG) sample template is chosen and transferred to the login. If needed, the requested tests are added. The sample identifications, date and time of collection, and date received are entered. The date on which the laboratory takes possession of the samples is defined as the date the COC is signed. For after-hours sample receipt where the after hours receipt form is used, the COC is not signed until the next day (see 5.1.11). If the collection time is not provided, a conservative estimate of the time is made. In cases where the client and project information is not in LIMS, SMO staff will setup the project with the available information on the COC.

5.2.3 Each test is automatically assigned a container ID in LIMS. The specific containers received are reconciled at the time of login.

5.2.4 The service request report is printed and a master Service Request File is created. Another person in the Sample Management group performs a login review. The master SR file is then given to the Project Chemist for final review before being issued to the laboratories. The Project Chemist reviews the service request form before being issued to the analysts. For analyses with short holding times (generally 48 hours and less), other samples near holding time expiration, and RUSH samples (due within 5 days), a "Temporary Service Request" is issued directly to the applicable department, whose representative signs off on receipt of the service request. SMO also prints a "2-day report" from LIMS that identifies all analyses with holding times expiring within 2 days and due dates within 2 days.

5.2.5 Lab Codes and Sample Identification/Labeling

Each sample is given a unique sample lab code by the LIMS system. This number is designated by the service request number, followed by a dash, a sequential number, a period and a container ID (e.g. K0504567-001.001), where K0504567 represents the service request number, -001 represents the sample and .001 represents the container. Thus, each sample is given a unique ID. Samples are reviewed to make sure the lab codes match the correct sample identification on the service request and that correct bottles were received for the analyses.

5.2.6 Solid Samples Labeling

5.2.6.1 If the quantity of sample given to the laboratory is insufficient for the analyses requested, the sample container should be labeled with Limited Sample Quantity (or something similar) or attach a "LIMITED SAMPLE VOLUME" tag. The project chemist will set the priorities for the analyses and, if possible, estimate the quantity of sample to be used for each test. This information should be documented and communicated to the lab via a Form V. For example, 8081 use 10 g; metals use 1 g; TCLP use 20 g.

NOTE. Samples that are quantity limited and require multiple analyses from a single container must be identified as soon as possible. Optimally, this should happen during sample login; however, discovery at any time should follow the same process. When it is discovered that a sample is quantity limited, the quantity of wet sample available is estimated and the project chemist is immediately notified. The project chemist should attempt to prioritize the tests most critical to the client (*i.e.*, which test should be performed first) and estimate the sample quantity allotted to each test.

5.2.6.2 Solid Samples Requiring VOC and Non-VOC Analyses with One Container

When only one container is received and both VOC and non-VOC analyses are requested, during sample receiving a sample custodian will either place a custody seal on the sample container on which is written ***VOC Analysis First*** (or something similar) or attach a "VOA FIRST" tag.

The VOC department will remove a sample aliquot first for the VOC analyses. The VOC department will handle the sample as if it were a Rush analysis, so that the other non-VOC analyses will not be unduly delayed. The VOC chemist who opens the container will either break the custody seal and will initial and date it when the container was opened or

sign and date the "VOA FIRST" tag. A ***VOC Analysis First*** note will also be included on the SR.

Sample aliquots for non-VOC analyses may not be taken from the sample container until the aliquot for the VOC analyses has been removed. Once the aliquot for the VOC analyses has been taken from the sample container, sample aliquots for the non-VOC analyses can be removed as prescribed in the various applicable analytical SOPs.

5.2.6.3 For solid samples requiring VOC and Non-VOC analyses with multiple containers, one of the sample containers will be designated for VOC analyses by either placing a custody seal or a "VOA FIRST" tag on the container.

5.3 Login Review

5.3.1 A peer review of the login and newly generated Service Request (SR) is done by the SMO staff. The SR is compared to the chain-of-custody to ensure that correct and complete information is entered on the SR. Refer to the SMO Login Review sheet (see Appendix).

In some cases (i.e. more complex projects) and at the Project Chemist's discretion, the SMO peer review may be performed by the Project Chemist. In this case, the review of the folder is documented by the Project Chemist when updating LIMS and/or initialing the service request.

5.3.2 As mentioned in section 5.2.3 above, the Project Chemist performs a final review of the login. At a minimum, this must include a close examination of the SR and chain-of-custody to verify that the SR generated by SMO accurately reflects the specifications for the project and samples. The Project Chemist may also need to refer to other project-related information, such as project QAPPs, client communication records, etc. If the SR does not accurately specify the work requirements, is incorrect, or is incomplete, the Project Chemist makes the needed changes and the draft SR file returned to SMO for revision. Once the SR is correct, the Project Chemist documents the review by signing the original SR.

5.4 Thermometer calibration and temperature measurement

5.4.1 The QA department performs thermometer calibration on all thermometers used to monitor sample storage temperature (see SOP ADM-TCAL). Each storage thermometer is given a unique numerical identification tag if one does not already exist, and all records of calibration are kept in the thermometer calibration notebook (SMO-TEMPCAL). The correction factor is recorded on the thermometer label.

5.4.2 Temperature measurement is performed for coolers using calibrated digital thermometers. The thermometer is plunged into the packing material, if possible, or placed as deep into the cooler as is practical and the lid closed. When coolers are packed such that samples are isolated from the main cooler compartment, via plastic bag and/or insulation (foam, vermiculite, etc.), place the thermometer down into the sample area. When multiple samples are in one large bag, place the thermometer in the bag. Do not just place thermometer on top of the bag or insulating material. The equilibration time is a minimum of 10 minutes.

5.4.3 The correction factor is applied to the temperature given by the thermometer after a measurement, and it is the corrected temperature that is recorded on the CRF. As an example, if the correction factor is -0.5, and the thermometer reads 5.9°C, then the corrected temperature is 5.4°C. If a temperature blank is present, the same thermometer is used to read the temperature blank when the cooler is unpacked. After measurement of the temperatures, all of the information is recorded in the appropriate space on the cooler receipt form.

Note: A temperature blank is generally considered more indicative of sample temperature inside the cooler. Where a temperature blank is present, its temperature reading is considered the *primary* temperature of the cooler and the second reading obtained from the thermometer placed in the cooler is for additional information.

5.5 Sample Storage, Security, and Internal COC

5.5.1 There are a number of refrigerators, freezers, and room temperature storage areas dedicated to specific storage requirements (e.g. Water VOCs, Soil VOCs, etc...) All of the sample storage facilities are located in the secured area. Samples are held in refrigerators until analysis is completed and reported to the client. Refrigeration of all non-tissue samples is maintained at a temperature of 4 $\pm$ 2° C unless otherwise specified by the state, project, or client specific protocols. Exception: Soil and sediment sample for designated for mercury analysis by EPA Method 1631 are stored frozen upon receipt. Tissue samples are stored in freezers at -16 to -24° C. Room temperature sample storage is specified in some cases.

Note: If a new or moved storage unit is to be placed in service, QA must be notified before using it so it can be monitored for acceptable conditions. QA will document and approve the unit for use prior to using for sample storage. Once SMO is notified, QA will begin regular monitoring.

5.5.2 When all samples are labeled accordingly with lab codes, they are placed in appropriate refrigerators in a secured area. This area is secured with combination locks which can be accessed by technical laboratory personnel, project managers, administrative support personnel and all senior staff members. Location of samples is recorded on the Service Request form and in internal COC records.

5.5.3 Internal COC procedures for checking samples in and out are described in SMO-SCOC SOP. The SMO department has a Sample Custodian who is responsible for retrieving/returning samples from/to storage for analysts. Analysts submit a requisition form to the Sample Custodian for the samples needed. Using the requisition form, the Sample Custodian pulls the specific sample bottles from storage and checks them out. The samples are loaded on a cart for the lab and the analyst is notified. When the analyst is finished using the samples, the cart is returned to the Sample Custodian with the requisition form. The Sample Custodian checks the samples back into custody/storage.

5.5.4 As an alternative to the Sample Custodian sample retrieval system, analysts may retrieve and check samples out/in directly themselves.

NOTE: Regardless of which procedure is used, the analyst is responsible for taking custody of the sample in LIMS, ensuring the internal chain of custody of samples is accurately documented. It is also the responsibility of the analyst to return samples to the storage area when work is completed, and relinquishing custody in LIMS.

5.5.5 Once the Analytical Report has been mailed to the client, the Internal Chain of Custody record from the bar-coding system database is printed and retained in the Service Request File.

5.6 Sample Disposal

After all analyses have been completed and the Certified Analytical Report (CAR) has been generated and mailed to the client, the associated samples are pulled from refrigeration and stored on shelves located in the secure storeroom area. An assessment is made of the nature of the samples (i.e. hazardous or non-hazardous) and the samples disposed of accordingly. Water samples are held for 31 days and soil samples for 2 months unless contractual obligations supersede these parameters. Fish and invertebrate animal tissue samples are held at -20°C for 90 days after reporting. After the holding period has expired, samples are disposed of using appropriate disposal procedures provided by the health and safety officer and/or committee. This task is performed by qualified SMO personnel.

5.7 Sample Shipping to Outside Labs

5.7.1 The sample is logged in and the specific product code, or "misc. outside", is used for subcontracted analysis. A list of routinely subcontracted analyses is attached.

5.7.2 For CAS Network Labs using LIMS, the samples are shipped with a chain of custody indicating the analyses to be performed.

5.7.3 For other labs, a purchase order (if non CAS lab) and chain of custody is filled out for work going to another subcontract laboratory. TAT, deliverables,etc. are specified. Prior to shipment, SMO personnel perform a review of the COC against samples subcontracted. Samples to be shipped to an outside lab are stored in a refrigerator near SMO.

5.7.4 A shipping form is completed by SMO and given to the shipping department for shipment.

5.7.5 Samples are prepared for shipping by the shipping department by packing in the appropriate packing material, placing blue ice, samples, and the chain of custody in shipping coolers. Custody seals are signed and dated and placed on the front and/or side of the cooler. The cooler is then sealed with packaging tape. A "Sample" label is placed on the cooler.

5.7.6 Samples that are relinquished to another laboratory that reports all of their data directly to the client and bills the client directly do not need to be logged in.

6. RECORDS

All sample receipt records, including COC forms, CRFs, Service Requests, sample information, etc. is filed in the master service request file.

Table 1

Minimum Sample Volume Requirements

Measurement	Matrix	
Inorganics	**Water (ml)**	**Soil (g)**
Color	50	--
Conductance	100	10
Hardness	100	--
pH	50	50
TSS	200	--
TDS	100	--
TS	100	10
TVS	100	10
Settleable Solids	1000	--
Turbidity	100	--
Total Metals	100	10
Metals w/reductive ppt.	1000	--
Dissolved Metals	100	--
Acidity	100	--
Alkalinity	100	--
Bromide	50	10
Chloride	50	10
Cyanide	500	10
Fluoride	50	10
Ammonia as N	50	10
TKN	500	10
Nitrate+Nitrite	100	10
Nitrate	50	10
Nitrite	50	10
Orthophosphorus	100	10
Total Phosphate	100	10
Sulfate	50	10
Sulfide	500	25
Sulfite	500	--
BOD	1000	50
COD	50	25
TOC	50	25
Total Phenols	500	25
MBAS	500	--
AOX/TOX	500	--

Table 1

Minimum Sample Volume Requirements
(continued)

Measurement	Matrix	
Organics	**Water (ml)**	**Soil (g)**
Oil and Grease	1000	25
DRO	500	30
Pesticides (8081A,8141)	1000	30
PCP (8151M)	40	30
PCBs (8082)	1000	30
PAHs (8270C, 8310)	1000	30
Semivolatiles (8270)	1000	30
GRO	2-40ml vials	30
BTEX (8021B)	2-40ml vials	30
Volatiles (8260B)	2-40ml vials	30
Dioxins	1000	30

Table 2

Sample Preservation Requirements

Inorganics pH Requirements

Analysis	Preservation requirement (pH)
Metals (total) except CrVI and Hg	<2
TOC	<2
COD	<2
Ammonia	<2
TKN	<2
Total Phosphorus	<2
Nitrate/Nitrite	<2
Sulfide	>9
Cyanide	>12
TOX/AOX	<2

Semivolatile Organics Residual Chlorine Requirements

Analysis	Requirement
Semivolatile Organics (625 and 8270)	None present
Organochlorine Pesticides (608 and 8081)	None present
Herbicides (8151)	None present

Appendix

Cooler Receipt and Preservation Form
Login Information Sheet
DC-1 Form
SMO Login Review Form

Columbia Analytical Services, Inc.
Cooler Receipt and Preservation Form

PC_____

Client / Project:_____ Service Request ***K08***_____

Received:_____ Opened:_____ By:_____

1. Samples were received via? ***US Mail*** ***Fed Ex*** ***UPS*** ***DHL*** ***GH*** ***GS*** ***PDX*** ***Courier*** ***Hand Delivered***
2. Samples were received in: (circle) ***Cooler*** ***Box*** ***Envelope*** ***Other***_____ ***NA***
3. Were custody seals on coolers? NA Y N If yes, how many and where?_____
   If present, were custody seals intact? Y N If present, were they signed and dated? Y N
4. Is shipper's air-bill filed? If not, record air-bill number:_____ NA Y N

5. **Temperature of cooler(s) upon receipt (°C):** _____ _____ _____ _____
   Temperature Blank (°C): _____ _____ _____ _____
6. If applicable, list Chain of Custody Numbers: _____ _____ _____ _____
7. Were custody papers properly filled out (ink, signed, etc.)? NA Y N
8. Packing material used. ***Inserts*** ***Baggies*** ***Bubble Wrap*** ***Gel Packs*** ***Wet Ice*** ***Sleeves*** ***Other***_____
9. **Did all bottles arrive in good condition (unbroken)?** *Indicate in the table below.* NA Y N
10. Were all sample labels complete (i.e analysis, preservation, etc.)? Y N
11. Did all sample labels and tags agree with custody papers? *Indicate in the table below* Y N
12. **Were appropriate bottles/containers and volumes received for the tests indicated?** NA Y N
13. Were the pH-preserved bottles tested* received at the appropriate pH? *Indicate in the table below* NA Y N
14. Were VOA vials and 1631 Mercury bottles received without headspace? *Indicate in the table below.* NA Y N
15. **Are CWA Microbiology samples received with >1/2 the 24hr. hold time remaining from collection?** NA Y N
16. Was C12/Res negative? NA Y N

Sample ID on Bottle	Sample ID on COC	Sample ID on Bottle	Sample ID on COC

Sample ID	Bottle Count	Bottle Type	Out of Temp	Head-space	Broken	pH	Reagent	Volume added	Reagent Lot Number	Initials

**Does not include all pH preserved sample aliquots received. See sample receiving SOP (SMO-GEN).*

Additional Notes, Discrepancies, & Resolutions:_____

Columbia Analytical Services Inc.
Cooler Receipt and Preservation Form

Client / Project: _____ Service Request *K08* _____

Sample ID on Bottle	Sample ID on COC	Sample ID on Bottle	Sample ID on COC

Sample ID	Bottle Count	Bottle Type	Out of Temp	Head-space	Broken	pH	Reagent	Volume	Lot Number	Initials

Additional Notes, Discrepancies & Resolutions: _____

COLUMBIA ANALYTICAL SERVICES, INC.
LOG-IN and RE-ISSUE INFORMATION SHEET

Date:_____

K_____

Sample ID's _____ _____

_____ _____

_____ _____

_____ _____

_____ _____

Received information for this request from:

_____ Project Chemist _____ Bottle Order _____ Client _____Bottles

_____ Others (Specify)_____

Re-issue Instructions: _____

_____

_____

_____

_____

Re-issue Due Date: _____

Special Instructions: _____

_____

_____

_____

SAMPLE LOG-IN SHEET

Nan Columbia Analytical Svc.	Page _____ of _____
Received By (Print Name)	Log-In Date
Received By (Signature)	
Case # / SDG Number	SAS Number

Remarks:		EPA Sample #	Corresponding Sample Tag#'s	Assigned Lab #	Remarks: Condition of samples, shipment, etc.
Custody Seal (s)	Present/Absent* Intact/Broken				
Custody Seal #'s	_____ _____				
Chain of Custody Records	Present/Absent*				
Traffic Report or Packing List	Present/Absent*				
Airbill	Airbill/Sticker				
Airbill #	_____ _____				
Samj s	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on COC				
Sample Condition	Intact/Broken/Leaking*				
Does information on custody records, traffic report and sample tags agree?	Yes or No				
Date received at Lab	_____				
Time received at Lab	_____				

Sample Transfer	
Fraction	Fraction
Area #	Area #
By	By
On	On
Contact SMO and attach record of resolution	
Reveiwed by	
Logbook #	
Logbook page #	

K08_____

Initial Login Review ☐ Revision Review ☐

Check the following information before the sample containers are shelved:

	Y	N	Is the CRF completed? (resolutions noted, etc.)
	Y	N	Are all bottles correctly labeled? (Client name vs. Lab code & number)
	Y	N	Is the Matrix consistent with the Chain of Custody (COC) and the bottles?
	Y	N	Are bottles labeled with the correct shelf number?
NA	Y	N	If indicated on the COC, are samples designated for QC specified on the SR?

Reviewed By:_____ Date:_____

Check the following information on the service request against the information on the COC:

NA	Y	N	Are sample names spelled correctly on the service request (SR)?
NA	Y	N	Is the date collected for each sample correct on the SR?
NA	Y	N	Is the date received correct on the SR? Multiple dates received?
NA	Y	N	Is the project name correct (as listed on the COC)? Discrepancies noted?
NA	Y	N	Is the project number correct (as listed on the COC)? Discrepancies noted?
NA	Y	N	Is the Tier level correct (as listed on the COC)? Discrepancies noted?
NA	Y	N	If listed on the COC, is the PO Number correct on the SR?
NA	Y	N	Are all the samples indicated on the COC logged in?
NA	Y	N	Are all analyses indicated on the COC logged in?
NA	Y	N	For RUSH TAT, does the due date listed on the SR make sense?
NA	Y	N	If any, are the pertinent comments on the COC indicated on the SR? (i.e. metals lists, volume limitations, hazards, etc.)
NA	Y	N	Has a temporary SR been issued for short hold-times and/or 24-48hr rushes?
NA	Y	N	If preservative was added upon receipt, is it indicated on the SR?
NA	Y	N	Does the SR contain appropriate additional product codes such as: EDD-DOD, Sample Prep, Filter codes, TS-Met?
NA	Y	N	Is the two- Day report printed?
NA	Y	N	Is the COC signed by SMO?

Reviewed By:_____ Date: _____

C-4

STANDARD OPERATING PROCEDURE

for

MERCURY IN WATER BY OXIDATION, PURGE AND TRAP, AND COLD VAPOR ATOMIC FLUORESCENCE SPECTROMETRY

SOP No.: MET-1631

Revision: 9

March 21, 2008

Approved by: _____ Supervisor — 3/21/08 Date

_____ QA Manager — 3-21-08 Date

_____ Laboratory Manager — 3/21/08 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

MERCURY IN WATER BY OXIDATION, PURGE AND TRAP, AND COLD VAPOR ATOMIC FLUORESCENCE SPECTROMETRY

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentration of Mercury in aqueous samples, including wastewater, aqueous wastes, and ground water, using EPA Method 1631E.

1.2. Method 1631 is a cold vapor atomic fluorescence procedure used in determining the total mercury (Hg) in filtered and unfiltered water samples. This method is designed for the measurement of total Hg in the range of 0.2 - 100 ng/L. With use of an additional calibration standard the range may be extended to 200 ng/L if necessary.

1.3. Total mercury as defined by this method means all BrCl-oxidizable mercury forms and species found in aqueous solution. This includes but is not limited to Hg(II), Hg(0), strongly organocomplexed Hg(II) compounds, adsorbed particulate Hg and several covalently bound organomercurials (i.e., CH_3HgCl, $(CH_3)_2Hg$, and $C_6H_5HgOOCCH_3$). The recovery of Hg bound within microbial cells may require the addition step of UV photo-oxidation.

2. METHOD SUMMARY

Samples are collected in new 500 mL Fluorinated LPE bottles. The samples are preserved with 1:1 HCl in the field, and with a BrCl solution at the lab. Samples are prepared for analysis by the addition of 5ml/L of BrCl solution or aliquoting 100 mL into pre-cleaned HDPE digestion vessels and oxidizing with BrCl solution. After oxidation the samples are pre-reduced with $NH_2OH{\cdot}HCl$ to destroy free halogens, and then reduced with $SnCl_2$ to convert Hg(II) to volatile Hg(0). Solid samples are prepared by HNO_3/ H_2SO_4 digestion than diluted with dilute BrCl solution to 40mls. An aliquot of 5ml is added to 100mls of prepurge water. The Hg(0) is separated from solution by purging with argon onto a gold trap. The trapped Hg is then thermally desorbed from the gold trap into an Ar gas stream that carries the Hg into the cell of the Brook Rand Model III cold-vapor atomic fluorescence spectrometer (CVAFS) for detection.

3. DEFINITIONS

3.1. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample analyses interspersed with calibration standards.

3.2. **Quality Control Sample (QCS)** - QCS solutions are made from a stock solution which is different from the stock used to prepare calibration standards and is used to verify the validity of the standardization.

3.3. **Matrix Spike** (MS) - In the matrix spike analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected.

3.4. **Matrix Spike Duplicate** (MSD) - In the matrix spike duplicate analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike duplicate is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the matrix spikes is calculated and used to assess analytical precision.

3.5. **Duplicate Sample** (DUP) - A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.6. **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure. In addition to method blanks, additional blanks are described within the procedure.

3.7. **Ongoing Precision Recovery (OPR)** - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.8. **Calibration Verification (VER)** – is spiked reagent water sample (aqueous blank spike) and is used to determine that the CVAFS remains in control.

4. INTERFERENCES

4.1. It is imperative that extreme care be taken to avoid contamination when collecting and analyzing ambient water samples for trace mercury. Potential sources of contamination during sampling include: metallic or metal-containing labware, containers, sampling equipment, reagents, and reagent water; improperly cleaned and stored equipment, labware, and reagents; and atmospheric inputs such as dirt and dust. Even human contact can be a source of trace mercury contamination (e.g., mercury fillings).

4.2. Within the laboratory, interferences from contaminated reagents must be minimized. Ultrex acids and reagent grade chemicals are used to prepare the reagents. To minimize contamination all sample preparation should be performed in the Class 100 clean hood. Before a given batch of samples is processed, all work surfaces in the hood or clean bench where the samples will be processed should be cleaned by wiping with a lint-free cloth or wipe soaked with reagent water.

4.3. All apparatus used for determination of mercury at ambient water quality criteria levels must be nonmetallic, free of material that may contain metals, or both.

4.4. Samples are taken in fluorinated LPE containers. These containers have a thin coating of a fluoropolymer.

4.5. It may be useful to dedicate separate sets of labware to different sample types; e.g., receiving waters vs. effluents. However, the apparatus used for processing blanks and standards must be mixed with the apparatus used to process samples so that any contribution of contamination from all labware can be randomly assessed.

4.6. Water vapor may collect in the gold trap and subsequently condense in the fluorescence cell upon desorption, giving a false peak due to scattering of excitation radiation. Condensation can be avoided by predrying the gold trap, and by discarding those traps that tend to absorb large quantities of water vapor.

4.7. Bottle blanks--After undergoing the cleaning procedures in this method, bottles should be subjected to conditions of use to verify the effectiveness of the cleaning procedures. A representative set of sample bottles should be filled with reagent water acidified to pH < 2 and allowed to stand for a minimum of 24 hr. After standing, the water should be analyzed for any signs of contamination. If any bottle shows signs of contamination, the problem must be identified, the cleaning procedures corrected or cleaning solutions changed, and all affected bottles re-cleaned. The is done through the preparation of method blanks weekly.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

5.4. Chronic mercury exposure may cause kidney damage, muscle tremors, spasms, personality changes, depression, irritability and nervousness. Organomercurials may cause permanent brain damage. Because of the toxicological and physical properties of the Hg, pure

standards should be handled only by highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks.

5.5. CAS purchases a dilute standard solution of Hg for this method. If primary solutions are prepared, they must be prepared in a hood.

5.6. Hands should be washed thoroughly after each manipulation and before breaks.

5.7. The cleanliness of work surfaces and tools may be assessed by wiping the surface with a piece of filter paper. Extraction and analysis by this method can achieve a limit of detection of less than 1 ng per wipe. Less than 0.1 mg per wipe indicates acceptable cleanliness; anything higher warrants further cleaning. More than 10 mg on a wipe constitutes an acute hazard and requires prompt cleaning before further use of the equipment or work space, and indicates that unacceptable work practices have been employed.

6. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

6.1. CAS laboratory staff does not collect samples. Samples are collected by field sampling staff of CAS customers using their sampling plans and procedures. In some cases, persons collecting samples may be required to be certified by regulatory bodies. Samples are either field- or laboratory-preserved by adding 5 mL of 6N (1:1) HCl to a 500 mL Fluorinated LPE bottle. Upon receipt at the laboratory, samples are taken to the clean room and BrCl (2.5ml/500ml) is added.

6.2. Samples that are acid-preserved only may lose Hg to coagulated organic materials in the water or the Hg may be condensed on the walls (Reference 19.11). The bottle should be vigorously shaken before sub-sampling to re-suspend the organic matter.

6.3. All handling of the samples in the laboratory should be undertaken in a mercury-free clean bench.

6.4. Water samples are typically stored at 4 ± 2°C until analysis. However, this is not a method requirement and samples may be stored at room temperature if necessary. The maximum holding time for mercury in unpreserved aqueous samples is 48 hrs. Once preserved, aqueous samples have maximum holding time of 90 days. Solid samples have a holding time of 1 year if stored in a freezer at < -15°C. Samples that are freeze dried have a holding time of 1 year, also.

6.5. Field blanks – Field blanks are used to demonstrate that samples have not been contaminated by the sample collection and transport activities.

6.5.1. Analyze the field blank(s) shipped with each set of samples (samples collected from the same site at the same time, to a maximum of 10 samples). Analyze the blank immediately before analyzing the samples in the batch.

6.5.2. If Hg or any potentially interfering substance is found in the field blank at a concentration equal to or greater than the ML (Table 1), or greater than one-fifth the level in the associated sample, whichever is greater, results for associated samples may be the result of contamination and may not be reported or otherwise used for regulatory compliance purposes.

6.5.3. Alternatively, if sufficient multiple field blanks (a minimum of three) are collected, and the average concentration (of the multiple field blanks) plus two standard deviations is equal to or greater than the regulatory compliance limit or equal to or greater than one-half of the level in the associated sample, results for associated samples may be the result of contamination and may not be reported or otherwise used for regulatory compliance purposes.

6.5.4. If contamination of the field blanks and associated samples is known or suspected, the laboratory should communicate this to the sampling team so that the source of contamination can be identified and corrective measures taken before the next sampling event.

6.6. Equipment blanks—Before any sampling equipment is used at a given site, equipment blanks (bottle blanks and sampler check blanks) must be submitted to the laboratory to demonstrate that the sampling equipment is free from contamination. Sampler check blanks are generated by processing reagent water through the sampling devices using the same procedures that are used in the field. Refer to Method 1631E for details.

7. APPARATUS AND EQUIPMENT

7.1. Cold vapor atomic fluorescence spectrometer (CVAFS): Brooks-Rand (Seattle, WA) Model III CVAFS, or equivalent.

7.2. Flowmeter, with needle valve capable of reproducibly keeping the carrier gas flow rate at 60 mL/min.

7.3. Pyrex bubbler with 4 way Teflon stopcock, 220 mL (Brooks-Rand, Seattle, WA, part no. AF-32 or equivalent)

7.4. Flow meter/needle valve capable of controlling and measuring gas flow rate to the purge vessel at 350 ± 50 mL/min.

7.5. Fluoropolymer fittings: Connections between components and columns are made using 6.4-mm o.d. fluoropolymer tubing and fluoropolymer friction-fit or threaded tubing

connectors. Connections between components requiring mobility are made with 3.2-mm o.d. fluoropolymer tubing because of its greater flexibility.

7.6. Acid fume pre-trap: 10-cm long x 0.9-cm i.d. fluoropolymer tube containing 2-3 g of reagent grade, non-indicating, ~14 mesh soda lime chunks, packed between wads of silanized glass wool. This trap must be cleaned of Hg by placing on the output of a bubbler containing reagent water and $SnCl_2$ and purging for 45 minutes with Ar.

7.7. Gold-coated sand trap or gold wire trap: 10-cm x 6.5-mm o.d. x 4-mm i.d. quartz tubing. The tube is filled with 3.4 cm of gold-coated 45/60 mesh quartz sand (Brooks Rand, Ltd., Seattle, WA, Part No. AF-20 or equivalent). The ends are plugged with quartz wool. A gold wire trap is also available from Brooks Rand (Part No. AF-19).

7.8. Traps are fitted with 6.5-mm i.d. fluoropolymer friction-fit sleeves for making connection to the system. When traps are not in use, fluoropolymer end plugs are inserted in trap ends to eliminate contamination. At least 16 traps are needed for efficient operation.

7.9. Heating of gold-coated sand traps: To desorb Hg collected on the traps, heat for 2-3 minutes to 450-500°C (a barely visible red glow when the room is darkened) with a coil consisting of 75 cm of 24-gauge Nichrome wire at a potential of 10 Vac. Potential is applied and finely adjusted with a variable transformer.

7.10. Air blower: After heating, the trap is cooled by blowing air from a small blower positioned immediately below the trap.

7.11. Computer (386 or better) and Windows Mercury Guru software to record and integrate the signal from the spectrometer.

7.12. Pipettors: All-plastic pneumatic fixed-volume and variable pipettors in the range of 20 uL to 1.0 mL.

7.13. Analytical balance capable of weighing to the nearest 0.001 g

7.14. Hot Block that is able to maintain temperature of 100 Celsius.

8. STANDARDS, REAGENTS AND CONSUMABLE MATERIALS

8.1. Document all reagent and acid preparation information in a logbook, including acids and acid mixtures. Label all reagents and acids/mixtures with appropriate identification,, tracking , and expiration date information.

8.2. Reagent water: Water in which mercury is not detected by this method; 18-megaohm ultra-pure deionized water starting from a pre-purified (laboratory DI water) source.

8.3. Argon: Grade 5.0 (ultra high-purity, GC grade) inert gas that has been further purified by the removal of Hg using a gold-coated sand trap.

8.4. Hydrochloric acid: trace-metal purified reagent HCl containing less than 5 pg/mL Hg.

8.5. Hydroxylamine hydrochloride: Dissolve 150 g of $NH_2OH{\cdot}HCl$ in reagent water and bring to 500 mL. This solution may be purified by the addition of 0.5 mL of $SnCl_2$ solution and purging overnight at 350 mL/min. with Hg-free Ar. Store tightly capped.

8.6. Stannous chloride: Bring 100 g of $SnCl_2{\cdot}2H_2O$ and 50 mL concentrated HCl to 500 mL with reagent water. Purge overnight with mercury-free Ar at 350 mL/min. to remove all traces of Hg. Store tightly capped.

8.7. Bromine monochloride (BrCl): Dissolve 5.4 g of reagent grade KBr in 500 mL of low-Hg HCl. Place a clean magnetic stir bar in the bottle and stir for approximately 1 hr in a fume hood. Slowly add 7.6 g reagent grade $KBrO_3$ to the acid with stirring. When all of the $KBrO_3$ has been added, the solution color should change from yellow to red to orange. Loosely cap the bottle, and allow to stir another hour before tightening the lid. KBr and $KBrO_3$ are purified by heating to 250°C overnight in an oven.

CAUTION: This process generates copious quantities of free halogens (Cl_2, Br_2, BrCl). which are released from the bottle. Add the $KBrO_3$ SLOWLY in a fume hood!

8.8. HNO_3/H_2SO_4 solution : In a fume hood, slowly add 300ml of concentrate H_2SO_4 to 700ml of concentrate HNO_3 in a Teflon bottle.

Warning : This mixture get hot and emits fumes.

8.9. 0.02 N Bromine monochloride solution: Dilute 100mls of concentrated BrCl solution to 1000ml with reagent water in a Teflon bottle.

8.10. Stock mercury standard: Stock mercury standards (1,000 ppm) are obtained from CPI International, Santa Rosa, CA, P/N 4400-1000331 and Inorganics Ventures, Inc., Lakewood, NJ, P/N CGHG1-5. A NIST-certified 10,000 ppm aqueous Hg solution (NBS-3133) is also available. These solutions are stable until the expiration date.

8.11. Secondary Hg standard: Dilute 0.100 mL of the stock solution (1,000 ppm) to 100 mL in water containing 1.0 mL of BrCl. This solution contains 1.00 ug/mL (1.00 ppm) Hg. Keep in a tightly closed fluoropolymer bottle. This expiration date is the same as the stock standard.

8.12. Working Hg standard A: Dilute 1.00 mL of the secondary Hg standard to 100 mL in a class A volumetric flask with reagent water containing 1.0% by volume BrCl solution. This solution contains 10.0 ng/mL and should be replaced monthly.

8.13. Working Hg standard B: Dilute 0.100 mL of the secondary Hg standard to 1000 mL with reagent water containing 1.0% by volume BrCl solution (10 mL BrCl/1000mL). This solution contains 0.1 ng/mL and should be replaced monthly.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Broad or asymmetrical peaks indicate a problem with the desorption train, such as low gas flow rate, water vapor on the trap(s), or an analytical column damaged by chemical fumes or overheating.

9.3. Damage to a trap is also indicated by a sharp peak, followed by a small, broad peak.

9.4. If the trap has been damaged, it and the fluoropolymer tubing downstream from it should be discarded because of the possibility of gold migration on downstream surfaces.

9.5. Gold traps should be tracked by unique identifiers so that any trap producing poor results can be quickly recognized and discarded.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. The acceptance criteria for test performance are listed in Table 1. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training and method proficiency.

11. PROCEDURE

11.1. An analytical batch is up to 20 field samples that are oxidized with the same reagents and analyzed within the same 12-hour shift. The analytical batch contains the following standards, samples, and blanks, in order:

Water Run:

Water blank 1
Water blank 2
Method blank 1
Method blank 2
5 ng/L opr and bubbler blank
5 ng/L QCS and bubbler blank
up to 10 samples
Matrix Spike
Duplicate Matrix Spike
OPR (optional)
Up to 10 additional samples
Matrix Spike
Duplicate Matrix Spike
Method blank 3
5 ng/L OPR and bubbler blank

Soil Run:

Calibration Verification (VER) and bubbler blank
Method Blank 1
Method Blank 2
OPR (use to demonstrate end to end analytical system)
QCS or SRM
up to 10 samples
Matrix Spike
Duplicate Matrix Spike
Calibration Verification (VER) and bubbler blank
Up to 10 additional samples
Matrix Spike
Duplicate Matrix Spike
Method blank 3
OPR (use to demonstrate end to end analytical system)
Calibration Verification (VER) and bubbler blank

For more than 20 samples in a 12 hour shift, the sequence is repeated.

11.2. Reagent blanks are analyzed in triplicate when there is a change in reagent(s). The average of these values is used to correct the sample reading for the contribution of the reagents to the measured mercury.

11.3. Sample Preparation

11.3.1. Record all sample preparation and sample information on the applicable benchsheet. This includes acid mixture tracking documentation.

11.3.2. Water samples may be preserved with 5ml/L BrCl, more may be required for complete preservation, an aliquot of 100mls may be added directly to bubbler. If more BrCl is used for the sample than 5ml/L, a method blank is prepared at the same level to be analyzed with that sample. If not, pour a 100-mL aliquot from a thoroughly shaken, acidified sample, into a 125-mL HDPE bottle. If estimated concentration is known, less sample may be used to keep the measured amount under 20ng (200 ng/L). Add bromine monochloride (BrCl), cap the bottle, and digest at room temperature for 12 hours. Shorter digestion times can be achieved by using elevated temperatures. For example, 70°C for 2 hours is adequate.

11.3.3. For clear water and filtered samples, add 0.5 mL of BrCl; for brown water and turbid samples, add 1.0 mL of BrCl. If the yellow color disappears because of consumption by organic matter or sulfides, dilute the sample. To minimize the contribution of background Hg, do not use more than 2.0 mL of the BrCl to oxidize the sample, rather dilute the sample until a permanent (12-hr) yellow color is obtained with 2.0 mL of BrCl or less.

11.3.4. Some highly organic matrices, such as sewage effluent, will require dilution as much as 1:10 and longer oxidation times, or elevated temperatures (i.e.; place sealed bottles in oven at 70°C for 2 hr). The oxidation must be continued until excess BrCl remains. This can be checked using starch-iodide paper.

11.3.5. Solid samples are prepared by weighing 400mg of sample into a 40ml vial. To each sample, add 10.0 ml of HNO_3/H_2SO_4 solution. A glass tapered marble placed over the digestion vessel. Samples are allowed to sit for 1 hour at room temperature. The samples are than placed in a Hot Block at 100° C for 2-3 hours refluxing. Once the samples have cooled they are diluted to 40 mL with 0.02 N BrCl solution. The samples then need to sit for At least 4 hours prior to analysis. This is to allow for complete oxidation of the methyl Hg.

11.3.6. Matrix spikes and matrix spike duplicates: For each batch of 10 or fewer samples, aliquot two additional 100-mL portions from a randomly selected sample, spike at the level specified in Section 12.3.3, and process in the same manner as the samples.

11.3.7. Method Blank: If samples are being preserved with BrCl, then three method blanks are prepared at the beginning of that week to represent the samples prepared that week. If samples weren't preserved with BrCl, than three method blanks are prepared for every 20, or fewer, samples by adding BrCl (0.5 mL) to 100 mL of

deionized water. The method blank uses bottles chosen at random from all sample bottles and serves as a check on the bottle washing procedure.

11.3.8. Water blank: At the same time as the samples are prepared, prepare a two water blanks by adding 0.5 mL of BrCl to 100 mL of DI water. The water blanks use bottles designated for this purpose and this serves as a check on the mercury content of the water and reagents. The mercury content of the water blank is used to correct the OPR, QCS, method blank values, and dilutions.

11.4. At the beginning of each 12 hour shift, attach lime traps to the bubbler and purge with Ar for 45-60 minutes to clean.

11.5. Instrument Initialization--Allow the instrument to warm-up for 30 minutes before attempting to analyze samples.

NOTE: *Purging of halogens onto the gold trap will result in damage and low or irreproducible results.*

11.6. Sample Analysis

11.6.1. Water Samples

11.6.1.1. Just prior to analysis, add 0.2 mL of NH_2OH to the BrCl-oxidized sample. Cap the bottle and swirl the sample. The yellow color will disappear, indicating the destruction of the BrCl. Allow the sample to react for 5 min. with periodic swirling to be sure that no traces of halogens remain.

11.6.1.2. Connect a lime trap and a gold trap to the bubbler, add 0.5 mL of $SnCl_2$ solution to the bubbler, pour the reduced sample into the bubbler, and purge the sample with Ar for 20 min. at 300-400 mL/min. At the end of the sparging time, remove the gold trap, plug the ends and save for analysis (See Section 11.7). Use the Bubbler blanks to correct the sample measurements and the water blank to correct the QCS, OPR, and Method Blank measurements. Remove the lime trap and rinse the sparging tube with deionized water. If necessary, use acetone to remove moisture from the upper portion of the sparging tube. It is important to make sure that the top of the sparging tube is dry for the next sample. Leave the nitrogen on while performing this procedure. Discard the water in the bubbler. Rinse the bottom half of the bubbler with deionized water. Close the nitrogen valve before adding the next sample to the bubbler.

11.6.1.3. Repeat 11.6.1.1 and 11.6.1.2 for each standard, blank, QC sample, and field sample.

11.6.2. Solid samples - A 100ml portion of water is pre-purged with 0.5ml $SnCl_2$ solution for 5 minutes. This is required for all samples and standards that are purged. Connect a lime trap and a gold trap to the bubbler. A VER is ran every ten samples and at the beginning and end of the run. A 5ml aliquot of the samples and standards are purged for 20 min. at 300-400 ml/min. They are sparged according to the run order (See Section 11.1). At the end of the sparging time, remove the gold trap, plug the ends and save for analysis (See Section 11.7). Use the Bubbler blanks to correct the sample measurements.

11.7. Desorption of Hg from the gold trap

11.7.1. Remove the plugs from the gold trap, place the Nichrome wire coil around the trap and connect it into the analyzer train between the incoming Hg-free argon and the detector.

11.7.2. Pass argon through the trap at a flow rate of approximately 30 mL/min. for 45-60 seconds to drive off condensed water vapor.

11.7.3. Apply electrical current (9.5-10 V DC) to the Nichrome coil around the gold trap and begin data collection. The applied electrical current will thermally desorb the Hg (as Hg(0)) from the trap into the detector.

11.7.4. After the 3-min. desorption time, stop data collection, turn off the current to the coil, and cool the trap (about one minute) using the cooling fan.

11.8. Place the next gold trap in line and proceed with analysis of the next sample.

Peaks generated using this technique should be very sharp and almost symmetrical. Mercury elutes at approximately 2 min. and has a width at half-height of about 11 seconds.

11.9. Calibration and standardization

11.9.1. The calibration must contain five or more non-zero points and the results of the analysis of 3 bubbler blanks. The lowest calibration point must be equivalent to the MRL, or lower.

11.9.2. Using the procedure in Section 11.7, standards are analyzed by the addition of aliquots of Hg working standard A (8.10) and Hg working standard B (8.11) directly into 100 mL of previously purged water in the bubbler. Add 0.5 mL and 2.0 mL of working standard B and 0.5 mL $SnCl_2$ to the bubbler. Swirl to produce a standard of 0.5 and 2.0 ng/L. Purge under the normal operating conditions described above. Sequentially follow with the addition of aliquots of 0.05, 0.20,

0.50, and 2.0 mL of working standard A plus 0.5 mL of $SnCl_2$ to produce standards of 5.0, 25.0, 50.0, and 200.0 ng/L.

11.9.3. For each point, subtract the mean peak area of the bubbler blanks for the batch from the area of each standard. Calculate the calibration factors (CF) for Hg in each of the six standards as follows:

$$CF = \frac{(C_s)}{(A_{Corr})}$$

where C_s = Concentration of the standard
A_{corr} = Bubbler blank corrected peak area

Calculate the relative standard deviation (RSD) of the calibration factor over the six-point range.

11.9.4. Calibration criteria are as follows:

(a) There must be a minimum of five non-zero calibration points.
(b) The difference between successive calibration points must be no greater than a factor of 10 and no less than a factor of 2 and should be approximately evenly spaced on a logarithmic scale over the calibration range.
(c) The relative standard deviation (RSD) of the calibration factors for all calibration points must be less than 15%.
(d) The calibration factor for any calibration point at a concentration greater than 100 ng/L must be within ±15% of the average calibration factor for the points at or below 100
(e) The calibration factor for any point <0.5 ng/L must be within 25% of the average calibration factor for all points.
(f) If calibration is to a higher range and this procedure is used for regulatory compliance, the ML must be less than one-third the regulatory compliance limit

11.9.5. Ongoing precision and recovery—Perform the ongoing precision and recovery test (12.3.1) to verify calibration prior to, and after in each analytical batch.

12. QA/QC REQUIREMENTS

12.1. Initial demonstration of capability

12.1.1. Laboratory performance is compared to the established performance criteria listed in Table 1. The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as follows:

12.1.2. Initial precision and recovery (IPR). To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

12.1.2.1. Analyze four replicates of the working Hg standard (Section 8.10) according to the procedure beginning in Section 11.3 – 11.7. These four replicates are prepared the same as samples are prepared.

12.1.2.2. Using the results of the set of four analyses, compute the average percent recovery (X), and the standard deviation of the percent recovery (s) for total Hg.

12.1.2.3. Compare s and X with the corresponding limits for initial precision and recovery in Table 1. If s and X meet the acceptance criteria, system performance is acceptable and sample analysis may begin. If, however, s exceeds the precision limit or X falls outside the acceptance range, system performance is unacceptable. Correct the problem and repeat the test.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples begins. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at or below the MRL) and analyze. Refer to the CAS *SOP for The Determination of Method Detection Limits and Limits of Detection*.

12.2.2. Calculate the average concentration found (x) in the sample concentration, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual, in the *SOP for Sample Batches*, and in method 1631E. An analytical batch is a set of samples oxidized with the same batch of reagents, and analyzed during the same 12-hour shift. A batch may be from 1 to 20 samples. Each batch must be accompanied by three method blanks, two OPR samples, and a QCS. In addition, there must be one MS/MSD pair analyzed for every 10 samples.

12.3.1. Ongoing precision and recovery (OPR): To demonstrate that the analysis system is in control and that acceptable precision and accuracy is being maintained within each analytical batch, the analyst shall perform the following operations:

12.3.1.1. Analyze the low-level Hg (5 ppt) working standard (Section 8.10) blank before and after analysis of each analytical batch according to the

procedure beginning in Section 11. Subtract the mean peak area of the bubbler blank and water blanks from the area for the standard and compute the concentration for the blank-subtracted standard.

12.3.1.2. Run a bubbler blank after each OPR and QCS sample. There must be at least 3 per analytical batch of 20 samples. See section 12.4.4.1.

12.3.1.3. Compare the concentration with the limits for ongoing precision and recovery in Table 1. If the concentration is in the range specified, the analysis system is in control and analysis of samples and blanks may proceed. If, however, the concentration is not in the specified range, the analytical process is not in control. Correct the problem and repeat the ongoing precision and recovery test.

12.3.2. Quality control sample (QCS): A QCS from a source different than the Hg used to produce the standards (OPR and working standards) must be analyzed at the beginning of each analytical batch. The acceptance criteria for the water QCS is 77-123%. For soils, a reference material routinely used and the acceptance criterion is the known concentration range provided with the material. If project-specified, a spiked blank matrix (Teflon beads) is used for the solid QCS, in which case the acceptance limits are 70-130%.

12.3.3. Matrix spike (MS) and matrix spike duplicate (MSD):

12.3.3.1. To assess the performance of the method on a given sample matrix, spike, in duplicate, a minimum of 10% (1 sample in 10) from a given sampling site or, if for compliance monitoring, from a given discharge. Blanks (e.g., field blanks) may not be used for MS/MSD analysis. A spike level of 5 ng/L has been found to acceptable for most samples.

Note: If, as in compliance monitoring, the concentration of Hg in the sample is being checked against a regulatory compliance limit, the spiking level shall be at that limit or at 1-5 times the background concentration in the sample, whichever is greater. If the sample concentration is not being checked against a regulatory limit, the spike shall be at 1-5 times the background concentration in the sample.

12.3.3.2. Spike two sample aliquots (MS and MSD) with the spiking solution and analyze these aliquots to determine the concentration after spiking (A).

12.3.3.3. Calculate the percent recovery (P) in each aliquot using the following equation:

100 (A-B)/T

where:
A = Measured concentration of analyte after spiking
B = Measured concentration of analyte before spiking
T = True concentration of the spike

12.3.3.4. Compare the percent recovery with the QC acceptance criteria in Table 1. If the results of spike fail the acceptance criteria, and recovery for the OPR standard for the analytical batch is within the acceptance criteria in Table 1, an interference may be present. The result may not be reported for regulatory compliance purposes. If the interference can be attributed to sampling, the site or discharge should be re-sampled. If the interference can be attributed to a laboratory error or deficiency, the analyst must take corrective action and repeat analysis of the associated samples (10 per MS/MSD pair) and MS/MSD.

12.3.3.5. If the results of both the spike and the OPR test fail the acceptance criteria, the analytical system is judged to be out of control. The analyst must identify and correct the problem and reanalyze the sample batch.

12.3.3.6. Relative percent difference between duplicates: Compute the relative percent difference (RPD) between the MS and MSD according to the following equation using the concentrations found in the MS and MSD. Do not use the recoveries calculated in Section 12.4.4.3 for this calculation because the RPD is inflated when the background concentration is near the spike concentration.

$$RPD = 200 \times (D1-D2)/(D1+D2)$$

Where:

Dl = concentration of Hg in the MS sample
D2 = concentration of Hg in the MSD sample

The RPD for the MS/MSD pair shall meet the acceptance criterion in Table 1. If the criterion is not met, the system is judged to be out of control. The problem must immediately be identified and corrected, and the analytical batch reanalyzed.

12.3.4. Blanks—Blanks are critical to the reliable determination of Hg at low levels. The sections below give the minimum requirements for analysis of blanks.

12.3.4.1.Bubbler blanks—Bubbler blanks are analyzed to demonstrate freedom from system contamination. At least three bubbler blanks must be run per analytical batch. One bubbler blank must be analyzed following each OPR.

12.3.4.1.1.Immediately after analyzing a sample for Hg, place a clean gold trap on the bubbler, purge and analyze the sample a second time using the procedure in Section 11, and determine the amount of Hg remaining in the system.

12.3.4.1.2.If the bubbler blank is found to contain more than 50 pg Hg, the system is out of control. The problem must be investigated and remedied, and the samples run on that bubbler must be reanalyzed. If the blanks from other bubblers contain less than 50 pg Hg, the data associated with those bubblers remain valid.

12.3.4.1.3.The mean result for all bubbler blanks (from bubblers passing the specification above) in an analytical batch (at least three bubbler blanks) is calculated at the end of the batch. The mean result must be < 25 pg with a standard deviation of < 10 pg for the batch to be considered valid.

12.3.4.1.4.If Hg in the bubbler blank exceeds the acceptance criteria, the system is out of control, and the problem must be resolved and the samples reanalyzed. Usually, the bubbler blank is too high for one of the following reasons:

- Bubblers need rigorous cleaning;
- Soda-lime is contaminated; or
- Carrier gas is contaminated.

12.3.4.2.Method Blank: At the same time as the samples are prepared, prepare three method blanks for every 20, or fewer, samples prepared by adding BrCl (0.5 mL) to 100 mL of deionized water. Four method blanks are prepared by adding 2.5ml/500ml in a clean sample bottles to represent samples that are preserved for that week. When samples require more BrCl for preservation than a method blank at the same level is prepared to accompany those samples for analysis. The method blank uses bottles chosen at random from all sample bottles and serves as a check on the bottle washing procedure.

12.3.4.3.Water blank: At the same time as the samples are prepared, prepare 2 water blanks by adding 0.5 mL of BrCl to 100 mL of DI water. The water blanks use bottles designated for this purpose and this serves as a check on the mercury content of the water and reagents. The mercury content of the

water blank is used to correct the OPR, QCS, and Method Blank values. One water blank is prepared at the beginning of purging samples.

12.4. Reagent blanks—The Hg concentration in reagent blanks must be determined on solutions of reagents by adding these reagents to reagent water in the bubbler.

12.4.1. Reagent blanks are required when the batch of reagents (bromine monochloride plus hydroxylamine hydrochloride) are prepared, with verification in triplicate for each new batch of reagents is needed.

12.4.2. Add aliquots of BrCl (0.5 mL), NH_2 OH (0.2 mL) and $SnCl_2$ (0.5 mL) to previously purged water in the bubbler. In order to evaluate the reagents as a potential source of contamination, the amount of reagent added to the reagent blank(s) must be the same as the amount of reagent added to the sample(s).

12.4.3. The presence of more than 20 pg of Hg indicates a problem with the reagent solution. The purging of certain reagent solutions, such as $SnCl_2$ or NH_2 OH with mercury-free argon can reduce Hg to acceptable levels. Because BrCl cannot be purified, a new batch should be made from different reagents and should be tested for Hg levels if the level of Hg in the BrCl solution is too high.

12.5. Bottle blanks—After undergoing the cleaning procedures in this Method, bottles should be subjected to conditions of use to verify the effectiveness of the cleaning procedures. A representative set of sample bottles should be filled with reagent water acidified to pH <2 and allowed to stand for a minimum of 24 h. Ideally, the time that the bottles are allowed to stand should be as close as possible to the actual time that the sample will be in contact with the bottle. After standing, the water should be analyzed for any signs of contamination. If a bottle shows contamination at or above the level specified for the field blank the problem must be identified, the cleaning procedures corrected or cleaning solutions changed, and all affected bottles re-cleaned.

12.6. As part of the QC program for the laboratory, method precision and accuracy for samples should be assessed and records maintained. Update the accuracy assessment regularly.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. It is the analysts responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in section 12.

13.2. Quantitation

13.2.1. Calculate the concentration of Hg in each sample directly from the mean calibration factor:

$$Hg(ng/L) = \frac{(A_s - \overline{A}_{BB}) \, x \, CF_m}{V}$$

where: A_s = peak area (or height) for Hg in sample
A_{BB} = peak area (or height) for Hg in bubbler blank (mean)
CF_m = Mean calibration factor
V = Volume of sample

13.2.2. Report results for Hg in reagent blanks separately.

13.3. Report results for samples in ng/L to two significant figures (three if >10 ng/L) for total Hg found above the MRL (See Table 1). Report results below the MRL but above the MDL as estimated values (J flagged). Report results below the MRL as ND, unless the project specifies reporting to the MDL.

13.4. If the result over the calibration range, prepare and analyze a diluted sample using the appropriate dilution factor to bring within range. For water samples, dilute the selected sample aliquot in reagent water and analyze. For soils, dilute an appropriate smaller aliquot of the soil digestate into reagent water and analyze. For samples far over range, an alternate procedure may be more appropriate. Consult with the Project Chemist before using another method.

13.5. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the CAS network directory R:\ICP\WIP. Once the results are transferred, the report is reviewed.

13.6. Refer to the *SOP for Laboratory Data Review Process* for general instructions for data review.

14. METHOD PERFORMANCE

14.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

14.2. The method detection limit (MDL) is established using the procedure described in the SOP for *The Determination of Method Detection Limits and Limits of Detection* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

15. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the *SOP for Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the *SOP for Report Generation* or in project-specific requirements.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18. TRAINING

18.1. Refer to the *SOP for Documentation of Training* for standard procedures.

18.2. Training outline

18.2.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.2.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.2.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

18.3. Training is documented following the *SOP for Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

19. REFERENCES

19.1. *Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry,* Method 1631, Revision E; USEPA, August 2002.

Table 1

Acceptance Criteria for Test Performance

Acceptance Criterion	Criteria
Method Detection Limit	0.08 ng/L[a]
Method Reporting Limit	1 ng/L[b]
Initial Precision and Recovery	
Precision (s)	± 21%
Recovery	79-121%
Interlaboratory Comparison	75-125%
Matrix Spike/Matrix Spike Duplicate	
Recovery	71-125%
Relative Percent Difference	± 24%
Ongoing Precision and Recovery	77-123%

a. Method 1631E states that the method detection limit has been determined to be 0.2 ng/L when no interferences are present.
b. Method 1631E states "the minimum level of quantitation (ML) has been established as 0.5 ng/L." The MDL and calibration can support the use of a 0.5 ng/L MRL if required by regulation or project protocols. With use of an additional calibration standard the range may be extended to 200 ng/L if necessary.

C-5

STANDARD OPERATING PROCEDURE

VOLATILE ORGANIC COMPOUNDS BY GC/MS

VOC-8260
Revision 12
March 21, 2008

Approved By:

Supervisor	3/21/08 Date
QA Manager	3.21.08 Date
Laboratory Manager	3/21/08 Date

COLUMBIA ANALYTICAL SERVICES, INC.
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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
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Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

VOLATILE ORGANIC COMPOUNDS BY GC/MS

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentration of volatile organic compounds in water and soil using USEPA Method 8260B. This method is also applicable to TCLP ZHE leachates and may also be applicable to various types of aqueous and nonaqueous waste samples.

1.2. Table 1 lists the compounds that can be determined by this method and the achievable method reporting limits (MRLs) in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstrated. The Method Detection Limits (MDLs) that have been achieved are given in Table 1, and may change slightly as MDL studies are repeated.

1.3. The nominal quantitation range for water samples is 0.5 – 80 ug/L. The nominal quantitation range for low concentration soils is 5-200 ug/kg. The nominal quantitation range for high concentration soils is 50-8000 ug/kg.

2. METHOD SUMMARY

2.1. This procedure gives gas chromatographic/mass spectrometric (GC/MS) conditions for the detection of parts per billion (ppb) levels of volatile organic compounds. A sample aliquot is injected into the gas chromatograph (GC) by either the purge and trap method or by direct injection. The compounds are separated on a fused silica capillary GC column. The compounds are detected by a mass selective detector (MSD), which gives both qualitative as well as quantitative information.

2.2. In the purge and trap process an inert gas, helium, is bubbled through the sample aliquot, at room temperature. This gas stream sweeps the volatile organic compounds out of the aqueous phase and into the gas stream - it purges the compounds out of the sample. The gas stream then passes through a sorbent column which selectively adsorbs, (traps) these compounds out of the helium. The preparation and analysis of soil samples uses procedures described in USEPA Method 5030B or 5035/5035A. After the purging sequence is done, the sorbent column (the trap) is heated and desorbed onto the GC column. The GC column separates the compounds and passes then onto the MSD for identification and quantification.

2.3. The sensitivity of this method depends on the level of background contamination (i.e. interferences) rather than on instrumental limitations. Highly contaminated waste samples will require a methanol extraction prior to analysis. This will elevate the reporting levels and may mask low levels of compounds of interest.

2.4. Deviations from the reference method(s): For water samples, a purge volume of 10mL is used, whereas the method (section 7.5.5) states 5 mL or 25 mL. The use of a 10 mL volume ensures sensitivity for "5 mL" type analyses *and*, on the analytical systems in use, meets the sensitivity goals of a 25 mL purge volume analysis. Also, the use of 10 mL rather than 25 mL decreases the negative effects of water being introduced into the P/T-GC-MS system.

3. DEFINITIONS

Analysis Window - Samples are analyzed in a set referred to as "a window". The window begins with the injection of the tune verification standard. After this standard has passed the method specific criteria a 12 hour analysis window is started. Next, a calibration curve or a continuing calibration standard (CCV see below) is run. If the CCV meets the specified criteria, sample and QC analyses are run until the 12 hour time limit closes. A new window must then be opened and the sequence repeated.

Internal Standards - Internal standards are organic compounds which are similar to the analytes of interest but which are not found in the samples. The chosen internal standards are used to help calibrate the instrument's response and to compensate for slight instrument variations from injection to injection.

Independent Calibration Verification (ICV) - Verification of the ratio of instrument response to analyte amount, a calibration check, is done by analyzing for analyte standards in an appropriate solvent. ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards.

Laboratory Control Sample (LCS) - In the LCS analysis, predetermined quantities of standard solutions of all analytes are added to a blank matrix prior to sample extraction and analysis. The purpose of the LCS is to monitor analytical control for the sample batch. Percent recoveries are calculated for each of the analytes.

Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of standard solutions of all analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

Standard Curve - A standard curve is a curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

Surrogate - Surrogates are organic compounds which are similar to the analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Continuing Calibration Verification Standard (CCV) - A mid-level standard injected into the instrument at specified intervals and is used to verify the initial calibration.

Method Blank (MB) - The method blank (also called continuing calibration blank) is a volume of clean reagent water analyzed on each GC/MS used for sample analysis. The purpose of the blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into other analyses.

4. INTERFERENCES

4.1. Interferences by common laboratory extraction solvents, such as Methylene Chloride, Acetone, and Freon 113 can cause problems. The area where volatile organic analyses are performed should be free of these solvents.

4.2. Other interferences include but are not limited to impurities in the inert purge gas, dirty plumbing/purge vessels, cross contamination by highly contaminated samples to clean ones in transport and storage, and carry over from one analysis to subsequent ones.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

6. SAMPLE CONTAINERS, COLLECTION, PRESERVATIONS, AND STORAGE

6.1. Refer to procedures for methods 5030 and 5035 for sample container and collection procedures. All sample containers for volatile organic analyses should be washed with soap and water, deionized water rinsed, and baked at 105°C ± 5°C for approximately 2 hours prior to use. Alternatively, one can buy precleaned sample containers from major lab equipment suppliers. All containers should be of glass or amber glass and equipped with a screw top cap and PFTE (teflon) lined septa.

6.2. Samples collected using EPA Method 5035 should be shipped in Encore sample tubes or collected in VOA vials containing sodium bisulfate (low concentration) and/or methanol (high concentration).

6.3. Collect all samples in duplicate, triplicate when possible. Prepare the proper number of sample bottles/containers prior to the sampling event with preservatives to adjust the samples pH to <2 with 1:1 HCI (water samples).

6.4. Slowly fill sample bottles to just overflowing taking care not to flush out the preservative or to entrain air bubbles in the samples. Seal the bottles with PFTE lined septa toward the sample and invert to check for entrained air bubbles.

6.5. Experimental evidence has shown refrigeration at 4°C alone will not stop biological degradation of some aromatic volatile organics. Adjusting the pH of the replicate samples to less than two (pH <2) with 1:1 HCl (@ 2-3 drops per 40 mLs) preserves samples for 14 days after collection. Residual chlorine can also degrade some organic compounds, generating trihalomethanes (THM's).

6.6. All samples must be stored at 4 ± 2°C and must be analyzed within 14 days of collection. See SOP VOC-5035 for additional holding time information. Any free product samples to be tested do not have any set holding times but should be analyzed as soon as possible.

7. APPARATUS AND EQUIPMENT

7.1. Gas chromatograph/Mass Selective Detector Systems

7.1.1. Each GC/MS system is set up with a GC capable of cooling the GC oven/column (subambient capability optional), injection onto a capillary column, and a stainless steel jet separator at the column's detector end prior to the transfer line interfaced with the MSD. Each MSD is a HP5970, HP5971, HP5972, or HP5973 that is controlled by the HP-MSDOS Chemstation software.

Instrument ID	Cofiguration	Column
MS02	Jet Separator	RTX-624, 75m, 0.53mm, 3um
MS04	Split/splitless – capillary direct	DB-624, 60m, 0.25mm, 1.4um
MS05	Split/splitless – capillary direct	RTX-624, 60m, 0.53mm, 1.8um
MS13, MS18, MS19	Split/splitless – capillary direct	RTX-624, 20m, 0.18mm, 1um
MS12	Split/splitless – capillary direct	DB-624, 20m, 0.18mm, 1.0um

7.1.2. Instrument systems and associated test methods are listed below. Individual operating conditions for 8260 instruments are given in Attachment A.

Instrument ID	Description	Tests Performed
MS02	5890/5970	8260W, 524.2, 624
MS04	5890/5972	8260W, 8260S, 524.2, 624
MS05	5890/5972	8260S
MS13	6890/5973	8260W, 8260S, 624
MS18	5890/5973	8260W
MS19	6890/5973	8260W, 8260S, 524.2
MS12	5890/5970	Screening only

7.2. Purge and Trap with Autosampler

Each volatile GC/MS analytical system uses a purge and trap to introduce the sample onto the GC column. A Tekmar LSC-2000 or its equivalent is needed. Each purge and trap has an autosampler (A/S) attached to run multiple samples, one at a time, and run unattended for extended periods of time. Varian Archon, Tekmar ALS 2016, or equivalents are preferred for extended unattended automated analyses.

7.3. GC Columns

- Restex RTX-624 (or equivalent) 75 M x 0.53mm id fused silica column 3.0µm film thickness
- Restex RTX-624 (or equivalent) 60 M x 0.32mm id fused silica column 1.8µm film thickness
- Restex RTX-624 (or equivalent) 20 M x 0.18mm id fused silica column 1.0µm film thickness
- J&W Scientific DB-624 (or equivalent) 60 M x 0.25 mm id fused silica column 1.4µm film thickness.
- J&W Scientific DB-624 (or equivalent) 20 M x 0.18 mm id fused silica column 1.0µm film thickness.

7.4. Each volatile GC/MS data processing station uses the most recent version of the EPA/NIST Mass Spectral Library. The current version is the NIST98k library.

7.5. Analytical balance - Capable of accurately weighing to 0.001g, Mettler PE160 or equivalent.

7.6. Syringes, Hamilton Gas-Tight in 10uL, 25uL, 100uL, 500uL, and 1000uL sizes.

7.7. Standard storage vials, screw thread with Mini-inert caps.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Methanol, purge and trap grade or equivalent.

8.2. Reagent water, prepared from deionized water, by charcoal filtration and then purging with high purity helium that is set at 4-5psi for approximately 2 hours prior to use.

8.3. Blank soil matrix – Ottawa sand, Accustandard specialty sands.

8.4. Helium, compressed high purity grade.

8.5. BFB Tuning Verification Stock Standard – A 25000ppm stock standard is purchased (Accustandard). This stock solution is diluted in methanol to give a working standard of 50ppm.

8.6. Stock Standard Solutions

8.6.1. Commercially prepared and certified stock standards are used routinely for all the method specified analytes. All such mixtures are also routinely checked against an independent source for both analyte identification and analyte concentration. All such stock standard mixtures have expiration dates given by the manufacturer and must be replaced if the comparison with the independent check standards indicates a problem. Alternatively, stock standards may be prepared from neat chemicals. Store with minimal headspace, at -10° to -20°C and protect from light.

8.6.2. When preparing stock standards from neat chemicals, accurately weigh approximately 0.1g of material and dilute with methanol to 10mL in a volumetric flask. If the purity of the neat chemical is <96% adjust the calculated concentration accordingly.

8.7. Working Standards - Prepare these standards from stock solutions. Prepare at concentrations which facilitate ease of preparation of instrument-level standards (calibration standards, etc.). Refer to Table 2 for Standard Expiration Date Guidelines. Store standards with minimal headspace in appropriately sized standard storage vials with mini-inert caps. Solutions should be checked for degradation or evaporation prior to use.

8.8. Calibration Standards

8.8.1. A minimum of five different concentration levels for all the analytes are prepared by diluting working standards into reagent water. The lowest concentration level must be at the method reporting level, or a level corresponding to a sample concentration meeting project-specific data quality objectives, with the remaining four levels defining the working linear range of the analytical system. *The permanent gas stock standards used to prepare calibration standards must not be more than one week old.* See Attachment B for detailed instructions and forms for preparing calibration standards and ICVs.

8.8.2. The suggested levels are 0.5, 2, 10, 20 and 40 ppb for waters; and 5, 20, 50, 100, and 200 ppb for soils. All calibration solutions are made up daily.

8.9. Internal Standards and Surrogates

The surrogates recommended are Dibromofluoromethane, toluene-d_8 and 4-bromofluorobenzene. The internal standards recommended are fluorobenzene, 1,4-difluorobenzene, 1,4-dichlorobenzene-d_4 and chlorobenzene-d_5. Other internal standards and surrogates may be used, depending on the analysis requirements. All surrogates and internal standards are added to every calibration standard. The spike level for samples, blanks, and matrix spikes is 10 ug/L for waters and 50 ug/L for soils.

8.10. Spiking Solutions

8.10.1. Prepare LCS and MS spiking solutions the same as ICV solutions as listed in Attachment B for calibration solutions. Use Level 6 for water spiking solution preparation and Level 3 for soil spiking solution preparation.

8.10.2. Matrix spike and laboratory control spike solutions should contain the full list of analytes of interest. However, a subset may be reported.

Note: Refer to Table 2 for Standard Expiration Date Guidelines.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Purge and Trap /Autosamplers

9.3.1. The purge/trap system should be baked out and back-flushed daily as needed, generally prior to use on a daily basis.

9.3.2. Replace the trap monthly, or sooner if performance deteriorates.

9.4. Gas Chromatograph

9.4.1. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.4.2. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

9.5. Mass Spectrometer

9.5.1. Tune the MS as needed to result in consistent and acceptable performance (see section 11).

9.5.2. For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

9.5.3. MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8260B, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water Samples

11.1.1.1. No preparation is generally required, other than dilution with reagent water to bring analytes into the upper half of the calibration range. Thus, a 10 mL sample volume is run straight from the sample vial. See the SOP for *Purge and Trap for Aqueous Samples* (VOC-5030) for details.

11.1.1.2. All water samples must be checked to have a pH $\leq$ 2 after sample analysis has taken place. Narrow range pH paper is used and the results are recorded on the injection log.

11.1.1.3. TCLP ZHE leachates are diluted 1:400 in reagent water prior to analysis. The TCLP samples and method blanks are diluted from the acidified ZHE extract; and the TCLP MS and LCS are diluted from a non-acidified extract, spiked, and poured into an HCL preserved VOA vial.

11.1.2. Soil samples are analyzed as either low concentration (direct purge) or high concentration (methanol preservation/extraction). Refer to the SOP for *Purge and Trap/Extraction for Volatile Organics in Soil and Waste Samples, Close System* (VOC-5035) for details.

11.1.2.1. For low concentration analyses, one of the sampling options given in method 5035 is to be used. Depending on the option used, follow the instructions given in the method. Typically, 1-5g is weighed out into the sample vial and 5 mL of reagent water is added. QC spikes and internal standards are then added, and the sample is purged at a temperature of 40°C° ±1°. Calibration standards, LCS, and method blanks require 5g Ottawa sand as the matrix.

11.1.2.2. In the event that low concentration analyses are specified but samples were not taken using a 5035 procedure, a portion of the sample is analyzed via direct heated purge of soil and EPA Method 5030A is cited. The analytical report should also be narrated with a statement indicating that 5030A has been deleted from SW-846. The low concentration analyses require a calibration specific to direct soil analysis.

11.1.2.3. The mid-level type is a methanol extraction method. In general, a 5g wet weight of soil is extracted with 5mL of purge-and-trap methanol in a scintillation vial. Place 5mL of purge-and-trap methanol into vial, tare, and add 5g of sample, and record the weight. Quickly cap and vortex until the sample is thoroughly mixed. A 1:100 dilution (500uL to 50mL) of this extract is then prepared in reagent water and analyzed using the water calibration. The extract weight, volume used, and methanol lot number are recorded on the injection log (or a bench sheet).

NOTE: For soil/solid samples requiring VOA and non-VOA analyses and only one container was submitted to the lab, sample receiving will label the sample container as "VOA Analysis First" and/or attach a "VOA FIRST" tag. The VOA department will remove a sample aliquot first for their analyses. The sample should be handled as if it were a Rush analysis, so that the other non-VOA analyses will not be unduly delayed. The VOA analyst who opens the container will either break the custody seal and will initial and date it when the container was opened or

sign and date the "VOA FIRST" tag. A VOA Analysis First note will also be included on the SR.

11.2. Calibration

NOTE: Refer to the CAS SOP *for Calibration of Instruments for Organics Chromatographic Analysis*. The calibration procedure(s) and options chosen must follow the CAS protocols. In general, the calibration procedure is as follows:

11.2.1. BFB Tuning

11.2.1.1. Prior to calibration and sample analyses, analyze a 25ng or 50ng injection of Bromofluorobenzene (BFB). Each volatile GC/MS analytical system set up to run 8260B must meet the criteria listed in Table 3 for the injection of BFB. The analysis time for BFB is used to define the start of the 12-hour window in which all analyses must be performed. Once the instrument is tuned, all subsequent analyses of standards, samples, and QA/QC samples within the same 12-hour window must be analyzed using the identical mass spectrometer operating conditions.

11.2.1.2. Obtain the spectrum for evaluation using one of the following options:

Note: The third option listed cannot be used for DoD QSM work

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the BFB peak or part of any other closely eluting peak.

- Use one scan at the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the BFB peak or part of any other closely eluting peak.

- Use one scan either directly preceding or following the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the BFB peak or part of any other closely eluting peak.

- Use the average across the entire peak up to a total of 5 scans. Peak integration must be consistent with standard operating procedure. If the peak is wider than 5 scans, the tune will consist of the peak apex scan and the two scans immediately preceding and following the apex. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the BFB peak or part of any other closely eluting peak.

11.2.1.3. Evaluate the spectrum against the criteria specified in Table 3. The criteria used must be the same for all ion abundance criteria checks associated with a given analysis. For example, initial calibration, continuing calibration(s), QC, and sample analyses for a given sample must all use the same criteria.

11.2.2. GC/MS Analytical System Initial Calibrations

11.2.2.1. Prior to conducting any sample analyses, a multi-point (5 point minimum) calibration must be run. Recommended calibration levels are 0.5, 2, 10, 20, and 40 ppb for waters, and 5, 20, 50, 100, and 200 ppb for soils. Analyze each calibration standard and tabulate the area response of the characteristic quantitation ions (Table 4) versus concentration for each compound, internal standards and surrogate. Calculate the response factors (RF) for each compound and surrogate relative to the specified internal standard by:

$$RF_x = \frac{(A_x)(C_{ISTD})}{(A_{ISTD})(C_x)}$$

Where:

A_x = Area of the characteristic quantitation ion for compound x.
A_{ISTD} = Area of the characteristic quantitation ion for the specified internal standard.
C_x = The concentration of the compound added.
C_{ISTD} = The concentration of the specified internal standard.

11.2.2.2. Calculate the mean response factor $(\overline{RF_x})$ for each analyte and surrogate from the five calibration levels. Calculate standard deviation (SD) and the percent relative standard deviations (%RSD) for each analyte from the mean with:

$$\%RSD = \frac{(SD)}{(\overline{RF_x})} 100.$$

11.2.2.3. The % RSD should be less than 15% for each compound. However, the %RSD for each individual CCC must be less than 30%. The CCC's are: 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene and Vinyl Chloride.

11.2.2.4. If a % RSD greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is required before performing recalibration.

11.2.2.5. If the % RSD for any compound is 15% or less, linearity can be assumed over the calibration range, and the relative response factor for each analyte and surrogate is used to quantitate sample analytes.

11.2.2.6. In those instances where the %RSD for one or more analytes exceeds 15%, the initial calibration may still be acceptable if the following conditions are met:

- The mean of the RSD values for all analytes in the calibration standards is $\leq 15\%$.
- The mean RSD criteria applies to all target analytes in the calibration standards, regardless of whether or not they are of interest for a specific project.
- The data user must be supplied with an initial calibration summary indicating the compounds which exceed 15% RSD and the result of the mean RSD calculation.

NOTE: Certain projects or programs, such as those under DoD QSM Version 3 and AFCEE QAPP 4.0, do not allow the use of the 'grand mean' (averaging of %RSDs). In these cases, use the QSM or QAPP-specified criteria and calibration options. If the grand mean is disallowed but no criteria is specified, use the Method 8000C criteria of RSD $\leq 20\%$ for all analytes.

11.2.2.7. If all of the conditions in Section 11.2.2.6 are met and the %RSD is less than 30%, then the average response factor may be used to determine sample concentrations as described in Section 13.1. If the %RSD exceeds 30%, then a linear regression or quadratic calibration is used.

11.2.2.8. The response of 5 SPCC's must also be checked for their minimum $\overline{RF_x}$:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chrorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

11.2.3. Independent Calibration Verification

11.2.3.1. Following initial calibration, analyze an ICV standard. The ICV solution must be obtained for all analytes that are analyzed and reported. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. The acceptance limits for the ICV are the same as those for the CCV. For DoD QSM projects, the criteria is within ± 25% of the expected value.

11.2.3.2. If a second source standard is not available or is cost prohibitive (such as certain non-routine analytes), then an independently prepared solution (prepared by analyst other than analyst preparing initial calibration standards) may be used as the ICV and must meet the criteria above.

11.2.3.3. After the multi-point calibration has passed all of the above criteria, and the Independent Calibration Verification has been performed, samples can be analyzed. The calibration curve mid-point standard may serve as the CCV for the opening set of samples within the same 12-hour window as the initial calibration.

11.2.4. Daily GC/MS Calibration

11.2.4.1. The start of a 12-hour analysis window requires a check of the instrument tune via an injection of 25ng or 50 ng of BFB. Refer to section 11.2.1 for the procedure. If the criteria found in Table 3 are met, then a check of the initial calibration curve is done. If the first analysis of the BFB fails, inspect the instrument for malfunction and perform maintenance as necessary. A second BFB tune verification may then be performed. If the second run also fails, it may be necessary to retune and recalibrate the system.

11.2.4.2. After the tuning criteria have been verified, the initial calibration must be checked and verified by analyzing a midrange calibration standard. The 10 ppb level for waters and 50 ppb level for soils is recommended. For water, CCVs are prepared by adding 10µl of the 50 ppm 8260 working standard and 5µl of the 2000 ppm ketone mix into 50 mL reagent water, and a 10 mL aliquot is purged (for MS02, the ketones are in the 50ppm 8260 working standard and the ketone mix is not needed). For soil, CCVs are prepared by adding 12.5µl of the 200ppm (nominal) working standard into 50 mL reagent water, and a 5 mL aliquot is purged. The results are compared with those of the initial calibration's $\overline{RF}$. The criteria for the SPCCs, as outlined above, must be met. Also, the CCCs must meet < 20% drift from the initial calibration curve average RFs. For non-CCC analytes, the maximum allowable %Dif or %Drift is ±30%, except dichlorofluoromethane, chlormethane, bromomethane, 2-Chloroethylvinylether, iodomethane, and vinyl acetate, which are ±40%.

NOTE: Certain projects or programs, including the DoD QSM, may require the use of project-specific criteria and evaluation options, including some found in Method 8260C and 8000C. In these cases, use the QSM or QAPP-specified criteria and calibration options.

11.2.4.3. If the tune criteria and the continuing calibration criteria are met, then the retention times of all compounds, surrogates, and internal standards are checked against the initial calibration. If the retention time for any internal standard changes by more than 30 seconds from the retention time from the mid-point standard of the most recent initial calibration, the system must be inspected for malfunctions and corrections must be made, as required. If the area for any of the internal standards changes by a factor of 2 (-50% to +100%) from the area from the mid-point standard of the most recent initial calibration, corrections must be made to the system.

11.2.5. Quantitation of all compounds will be based on the initial calibration.

11.3. Identification of Analytes

The MSD data system software identifies a sample component by first finding and identifying the surrogate and internal standards. After they have been integrated, the extracted ion chromatogram is searched for all calibrated analytes. The data system makes a positive identification for any peak associated with the proper time window and having the primary and secondary ions (as specified in the data system "method" file) in characteristic relative abundances. The analyte result is then calculated. The analyst reviews all analyses to confirm (or correct) all data system qualitative interpretations. The analyst should follow interpretation guidelines in the method for identification of analytes. Table 4 lists characteristic ions as given in Method 8260B. If there is no peak found for an analyte in the expected retention time window and the mass spectrum does not match according to the method criteria, then the analyte is "not found". Print out spectra for all confirmed hits.

11.4. If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample, using the SOP for Total Solids.

12. QA/QC REQUIREMENTS

Note: The analyst should refer to the CAS *SOP for Sample Batches* and the CAS *SOP for The Determination of Method Detection Limits and Limits of Detection.*

12.1. Initial Precision and Recovery Validation

The accuracy and precision of the procedure must be validated before analysis of samples begin, or whenever significant changes to the procedures have been made. To do this, analyze four water sample spikes, calculate the average recovery and standard deviation, and evaluate as described in EPA SW-846. The concentration of the analytes to be spiked should be in the

working calibration range. Initial Precision and Recovery studies should be performed as part of analyst training. Copies of the studies should be maintained in the lab and in the analyst's training file.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples begins. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the CAS SOP for The Determination of Method Detecion Limits.

12.2.2. Calculate the average concentration found (x) in the sample concentration, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the *SOP for Sample Batches*. For every sample batch, at least one method blank and one LCS, and one MS/DMS pair must be run for each matrix. Analytical windows must have at least one base (primary) sample analysis to require a MS/DMS pair.

12.3.1. Method blank - A method blank is analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must also be run every 12-hour window to demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank. For some project specific needs, additional requirements or exceptions may be given.

Note: For DoD projects, no target analytes should be detected in the Method Blank above ½ the MRL, or above the MRL for common lab contaminants. Methylene chloride and acetone are the common lab contaminants.

12.3.2. A lab control sample (LCS) must be prepared and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix spike solution, and going through the entire preparation and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X =Concentration of the analyte recovered
TV =True value of amount spiked

Evaluate the recovery using the QC acceptance criteria in Attachment C or project-specific acceptance limits. If the lab control sample (LCS) fails acceptance limits for any of the control compounds, any associated sample data is rejected and the analyst must take corrective action. Corrective action includes an evaluation of the sample preparation, analytical system, and calibration; and may require re-extraction, re-analysis, and/or recalibration and re-analysis.

12.3.3. A matrix spike/duplicate matrix spike (MS/DMS) must be prepared and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire preparation and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where: X = Measured concentration of the spiked sample aliquot
X1 = Measured concentration of unspiked sample aliquot
TV = True value (theoretical concentration) of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where: R1 = measured concentration of the first sample aliquot
R2 = measured concentration of the second sample aliquot

Evaluate the recovery and RPD using the QC acceptance criteria in Attachment C or project-specific acceptance limits*. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken.

* For DoD project, matrix spike recoveries are compared to LCS acceptance limits.

12.3.4. Calculate and evaluate the surrogate recovery using the QC acceptance criteria in Attachment C. If surrogate recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be identified.

12.4. Acceptance Criteria

12.4.1. The acceptance criteria for tuning verification, initial, and continuing calibration verification have been outlined above in Section 11.

12.4.2. Current acceptance criteria for matrix spikes, LCSs and surrogates are listed in Attachment C and in QA document QCLIMITS.xls. The acceptance criteria listed are current criteria, but are subject to change as control limits are updated.

12.5. Corrective action requirements have been outlined above in Section 11. Also, the corrective action requirements of any project-specific project plan should be used when applicable.

12.6. Additional QA/QC measures include control charting of QC sample results.

13. DATA REDUCTION , REVIEW, AND REPORTING

13.1. Calculations

13.1.1. The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions. The results for a water sample are calculated as follows when $\overline{RF_x}$ is used:

$$A_x = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where: A_x = the amount, in ppb, of the analytes in the sample;
$Resp_x$ = the peak area of the analytes of interest;
$Resp_{ISTD}$ = the peak area of the associated internal standard;
Amt_{ISTD} = the amount, in ppb, of internal standard added

$\overline{RF_x}$ = the average response from the five-point for the analytes of interest.

13.1.2. The results for low concentration soil work are calculated by taking the normal print out, in ppb, (see the water results outlined above) and correcting for the total, dry soil sample actually purged:

$$(A_x) = \frac{(5\ grams)}{(ASW_t\ gr)(\%\ Solids)} = A_x\ Low\text{-}Level\ Soil$$

Where: A_x = the amount, in ppb, from the data system; five grams is the nominal amount of soil that is heated and purged;
ASW_t = the actual soil wet weight, in grams, that is purged; and
% Solids the correction factor for dry weight.

13.1.3. Results for a high concentration soil samples (methanol extracts) are calculated as follows:

$$(A_x) = \frac{(Dilution)(V_{EXTR})}{(ASW_t)(\%\ Solids)} = A_x\ High\text{-}Level\ Soil\ Amt.$$

Where: A_x = the data station results, in ppb;
Dilution = the dilution of the extract.
ASW_t = the actual wet weight of soil extracted; and
% Solids = the dry soil correction.
V_{EXTR} = the methanol extract volume (mL)*

* The water contained in the native sample is accounted for when determining the final extract volume. The final volume of the methanol extract is the total volume of the methanol/water mixture. Calculate the final volume as follows:

$$Final Volume\ Methanol/Water = mL\ of\ solvent + \left(\frac{\%Moisture\ x\ Sample\ Wt.(g)}{100} \right)$$

13.2. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* for details.

13.3. Reporting

13.3.1. Reports are generated using the STEALTH Data Reporting System which compiles the SMO login information and Enviroquant data. This compilation is then transferred to a file, which STEALTH uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.3.2. Alternatively, Excel templates located in R:\VOA\forms may be used to prepare reports from hard-copy data. The analyst should choose the appropriate form and QC pages to correspond to required tier level. The detected analytes, surrogate and matrix spikes are then transferred, by hand, to the templates.

14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the *SOP for Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for Report Generation or in project-specific requirements.

15. METHOD PERFORMANCE

This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

The method detection limit (MDL) is established using the procedure described in the SOP for *The Determination of Method Detection Limits and Limits of Detection* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

18. TRAINING

18.1. Training Outline

18.1.1. Review literature by reading references. Review the EPA methodology and any applicable state-specific methods. Review the SOP. Also review the MSDS for methanol.

18.1.2. Observe the procedure as performed by an experienced analyst at least three times.

18.1.3. Assist in the procedure under the guidance of an experienced analyst for a period of three months. During this training process, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.1.4. Following the three-month training period the analyst is expected to complete an initial precision and recovery (IPR) study for solid samples by direct, solid samples by extraction, and water samples. Summaries of the IPR are reviewed and signed by the supervisor and forwarded to the employee's training file.

18.1.4.1. Perform IPR studies by preparing and analyzing four replicate laboratory control samples spiked at a level of 10-20 times the MRL. Calculate average percent recovery and relative standard deviation for the four replicate analyses. Refer to Method 8000B and 8260B for analysis and evaluation guidelines.

18.1.4.2. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

18.2. Training is documented following the SOP for Documentation of Training.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

19. REFERENCES

19.1. CAS SOP for Purge and Trap for Aqueous Samples (VOC-5030).

19.2. CAS SOP for Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples, Closed System (VOC-5035).

19.3. *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique*, U.S. EPA, SW-846, Final Update III, Method 8260B, Revision 2, December 1996; and Method 8260C, August 2006.

19.4. *Purge and Trap*, U.S. EPA, SW-846, Final Updates I and III, Methods 5030A (Rev. 1), 5030B (Rev. 2), and 5030C, May 2003.

19.5. *Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste*, U.S. EPA, SW-846, Final Update III, Method 5035, Revision 0, December 1996; and Method 5035A, July 2002.

TABLE 1
Method 8260B Analyte List

Compound	Water		Soil – Low (wet)		Soil – High (wet)	
	MRL (ug/L)	MDL (ug/L)	MRL (ug/kg)	MDL (ug/kg)	MRL (mg/kg)	MDL (mg/kg)
1,1,1,2-Tetrachloroethane	0.5	0.111	5.0	0.51	.05	0.0111
1,1,1-Trichloroethane (TCA)	0.5	0.116	5.0	0.57	.05	0.0111
1,1,2,2-Tetrachloroethane	0.5	0.138	5.0	0.73	.05	0.0138
1,1,2-Trichloroethane	0.5	0.138	5.0	0.69	.05	0.00992
1,1-Dichloroethane	0.5	0.101	5.0	0.78	.05	0.00906
1,1-Dichloroethene	0.5	0.122	5.0	0.69	.05	0.0119
1,1-Dichloropropene	0.5	0.150	5.0	0.73	.05	0.0128
1,2,3-Trichlorobenzene	2	0.326	20	0.90	0.2	0.0326
1,2,3-Trichloropropane	0.5	0.213	5.0	0.61	.05	0.0213
1,2,4-Trichlorobenzene	2	0.218	20	0.77	0.2	0.0218
1,2,4-Trimethylbenzene	2	0.141	20	0.82	0.2	0.0141
1,2-Dibromo-3-chloropropane	2	0.991	20	0.85	0.2	0.0991
1,2-Dibromoethane (EDB)	2	0.0981	20	0.79	0.2	.00730
1,2-Dichlorobenzene	0.5	0.111	5.0	0.65	.05	0.00847
1,2-Dichloroethane (EDC)	0.5	0.114	5.0	0.67	.05	0.0114
1,2-Dichloropropane	0.5	0.139	5.0	0.72	.05	0.0124
1,3,5-Trichlorobenzene	0.5	0.343	-	-	.2	0.0343
1,3,5-Trimethylbenzene	0.5	.121	20	0.82	0.2	.0121
1,3-Dichlorobenzene	0.5	0.102	5.0	0.71	.05	0.0102
1,3-Dichloropropane	0.5	0.147	5.0	0.52	.05	0.00759
1,4-Dichlorobenzene	0.5	0.114	5.0	0.82	.05	.00869
1,4-Dioxane	100	23.9	250	110	25	10
1-Chlorohexane	0.5	0.118	5.0	0.69	.05	0.0118
2,2-Dichloropropane	0.5	0.174	5.0	0.81	.05	0.0174
2-Butanone (MEK)	20	1.94	20	8.4	2.0	0.322
2-Chloroethyl Vinyl Ether	5	0.333	10	1.0	0.5	0.0112
2-Chlorotoluene	2	0.111	20	0.73	0.2	0.0111
2-Hexanone	20	3.96	20	6.1	2.0	0.396
2-Nitropropane	5	1.99	20	3.8	0.5	0.0238
3-Chloro-1-propene	5	0.154	-	-	0.5	0.0127
4-Chlorotoluene	2	0.118	20	.74	0.2	0.00884
4-Isopropyltoluene	2	0.128	20	0.72	0.2	0.0128
4-Methyl-2-pentanone (MIBK)	20	2.70	20	5.5	2.0	0.280
Acetone	20	4.08	20	10	2.0	0.229
Acetonitrile	50	7.44	100	69	0.5	0.105

TABLE 1
Method 8260B Analyte List

Compound	Water		Soil – Low (wet)		Soil – High (wet)	
	MRL (ug/L)	MDL (ug/L)	MRL (ug/kg)	MDL (ug/kg)	MRL (mg/kg)	MDL (mg/kg)
Acrolein	20	1.42	100	14	2.0	0.142
Acrylonitrile	5	0.531	20	3.2	2.0	0.0161
Benzene	0.5	0.136	5.0	0.79	.05	0.0105
Bromobenzene	2	0.172	5.0	0.81	0.2	0.00972
Bromochloromethane	0.5	0.164	5.0	0.52	.05	0.0126
Bromodichloromethane	0.5	0.109	5.0	0.53	.05	0.00847
Bromoform	0.5	0.279	5.0	0.65	.05	0.0279
Bromomethane	0.5	0.217	5.0	0.8	.05	0.0217
Carbon Disulfide	0.5	0.159	5.0	1.5	.05	0.0159
Carbon Tetrachloride	0.5	0.139	5.0	0.60	.05	0.0123
Chlorobenzene	0.5	0.134	5.0	0.70	.05	0.00933
Chloroethane	0.5	0.226	5.0	0.78	.05	0.0173
Chloroform	0.5	0.136	5.0	0.57	.05	0.00958
Chloromethane	0.5	0.136	5.0	0.99	.05	0.0136
Chloroprene	10	0.348	20	3.3	1.0	0.0119
cis-1,2-Dichloroethene	0.5	0.116	5.0	0.83	.05	0.0116
cis-1,3-Dichloropropene	0.5	0.110	5.0	0.76	.05	0.00808
cis-1,4-Dichloro-2-butene	10	0.600	20	14	1.0	0.0562
Cyclohexane	1.0	0.200	5.0	0.67	0.10	0.0200
Dibromochloromethane	0.5	0.104	5.0	0.60	.05	0.00817
Dibromomethane	0.5	0.119	5.0	0.72	.05	0.00974
Dichlorodifluoromethane	0.5	0.166	5.0	0.70	.05	0.0166
Dichlorofluoromethane (CFC 21)	0.5	0.400	-	-	.05	0.05
Diisopropyl Ether	2.0	0.244	10	0.25	-	-
Ethyl Acetate	5	0.618	20	4.1	0.5	0.0484
Ethyl Ether	1	0.5	-	-	0.1	0.1
Ethyl Methacrylate	5	0.0934	20	3.0	0.2	0.00934
Ethylbenzene	0.5	0.130	5.0	0.57	.05	0.00974
Ethylene Oxide	10	1.1	100	18	-	-
Hexachlorobutadiene	2	0.28	20	0.75	0.2	0.0380
Iodomethane	5	0.375	20	4.0	0.5	0.0224
Isobutyl Alcohol	100	5.01	200	94	50.	0.427
Isopropylbenzene	2	0.105	20	0.68	0.2	0.00680
m,p-Xylenes	0.5	0.219	5.0	1.5	.05	0.0186
Methacrylonitrile	5	0.444	20	3.4	0.5	0.0155
Methyl Acetate	1.0	0.354	5.0	0.73	0.10	0.0297

TABLE 1
Method 8260B Analyte List

Compound	Water		Soil – Low (wet)		Soil – High (wet)	
	MRL (ug/L)	MDL (ug/L)	MRL (ug/kg)	MDL (ug/kg)	MRL (mg/kg)	MDL (mg/kg)
Methyl Methacrylate	5	0.354	20	3.3	0.5	0.0118
Methyl tert-Butyl Ether	0.5	0.197	5.0	0.64	.05	0.00734
Methylcyclohexane	1.0	0.190	5.0	0.71	0.10	0.0190
Methylene Chloride	2	0.193	10	0.96	0.2	0.0193
Naphthalene	2	0.285	20	0.89	0.2	0.0285
n-Butylbenzene	2	0.221	20	0.75	0.2	0.0221
n-Hexane	1	0.180	10	4.0	0.1	0.0180
n-Octane	5	0.29	10	4.0	0.1	0.1
n-Propylbenzene	2	0.0980	20	0.72	0.2	0.00968
o-Xylene	0.5	0.102	5.0	0.69	.05	0.00785
Propionitrile	5	1.27	20	3.7	0.5	0.0862
sec-Butylbenzene	2	0.127	20	0.74	0.2	0.0127
Styrene	0.5	0.0943	5.0	0.73	.05	0.00943
tert-Amyl Methyl Ether	2.0	0.143	10	0.15	-	-
tert-Butyl Alcohol	20	1.031	50	3.6	0.25	0.103
tert-Butyl Ethyl Ether	2.0	0.0741	10	0.084	0.05	0.005
tert-Butylbenzene	2	0.122	20	0.74	0.2	0.0122
Tetrachloroethene (PCE)	0.5	0.126	5.0	0.31	.05	0.0109
Tetrahydrofuran	10	0.456	-	-	1.0	0.5
Toluene	0.5	0.108	5.0	0.84	.05	0.00975
trans-1,2-Dichloroethene	0.5	0.143	5.0	0.73	.05	0.0139
trans-1,3-Dichloropropene	0.5	0.0894	5.0	0.60	.05	0.00863
trans-1,4-Dichloro-2-butene	10	0.597	20	1.5	1.0	0.0597
Trichloroethene (TCE)	0.5	0.133	5.0	0.28	.05	0.0188
Trichlorofluoromethane	0.5	0.131	5.0	0.73	.05	0.0131
Trichlorotrifluoroethane	0.5	0.132	5.0	0.74	.05	0.0121
Vinyl Acetate	5.0	0.663	20	3.6	0.5	0.0387
Vinyl Chloride	0.5	0.042	5.0	0.62	.05	0.0211

TABLE 2

Standard Expiration Date Guidelines

Standard	Expiration time
Neat Chemicals	Expiration date 3 years from date opened, or supplier's assigned date.
Stock Standards (unopened ampules, commercially prepared or lab prepared)	Supplier's assigned date, or 1 year if no expiration date provided.
Opened ampules and working standards	
• concentration $\geq$ 5000 ppm	6 month expiration date.
• concentration 1000 - 5000 ppm	2 month expiration date.
• concentration 200 - 1000 ppm	1 month expiration date.
• concentration < 200 ppm	7 day expiration date.
Internal Standard Solutions	One month expiration date.

Note: The analyst performing specific analytical procedures should use judgement and take into consideration the solution reactivity, volatility, and concentration when using standards to prepare calibration curves. Certain standards, depending on use and storage, may have shorter usable life than described in these guidelines.

TABLE 3

4-Bromofluorobenzene Characteristic Ion Abundance Criteria

Mass	Ion Abundance Criteria **MS04, MS05, MS12**	Ion Abundance Criteria **MS02**	Ion Abundance Criteria **MS13**
50	15-40% of mass 95	15-40% of mass 95	15-40% of mass 95
75	30-60% of mass 95	30-80% of mass 95	30-60% of mass 95
95	Base peak, 100% relative abundance	Base peak, 100% relative abundance	Base peak, 100% relative abundance
96	5-9% of mass 95	5-9% of mass 95	5-9% of mass 95
173	< 2% of mass 174	< 2% of mass 174	< 2% of mass 174
174	> 50% of mass 95	> 50% of mass 95	> 50% of mass 95
175	5-9% of mass 174	5-9% of mass 174	5-9% of mass 174
176	95 -101% of mass 174	95 -101% of mass 174	95 -101% of mass 174
177	5-9% of mass 176	5-9% of mass 176	5-9% of mass 176
Reference:	EPA 8260B	EPA 524.2	EPA 8260B

TABLE 4
Characteristic Masses (m/z) for Purgeable Organic Compounds

Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64	66
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75

TABLE 4

Characteristic Masses (m/z) for Purgeable Organic Compounds (continued)

Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53
b-Propiolactone	42	43, 44

TABLE 4

Characteristic Masses (m/z) for Purgeable Organic Compounds (continued)

Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Surrogates:		
1,2-Dichloroethane-d4	65	67, 51
4-Bromofluorobenzene	95	174, 176
Dibromofluoromethane	113	111, 192
Toluene-d8	98	99, 70
Internal Standards:		
1,4-Difluorobenzene	114	63, 88
Fluorobenzene	96	77, 70, 50
1,4-Dichlorobenzene-d4	152	115, 150
Chlorobenzene-d5	117	119, 82

ATTACHMENTS

Attachment A
Instrument Operating Parameters

Attachment B
Detailed Standard Preparation Instructions

Attachment C
QC Acceptance Criteria

ATTACHMENT A

Method Information For: D:\MS02\METHODS\MS28260J.M

Method Sections To Run:

( ) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
(X) Post-Run Cmd/Macro = MACRO "loctonet.MAC",G

Method Comments:
8260 METHOD FOR SOIL

END OF TOPLEVEL PARAMETERS

ACQUISITION PARAMETERS

General Information

Target : GC
Tune File : JON.U
Acquisition Mode : Scan

MS Information

Solvent Delay : 2.10 min

EM Absolute : False
EMV Offset : 0.0
Resulting Voltage : 1458.9

[Scan Parameters]

Low Mass : 35
High Mass : 300
Threshold : 500
Sampling # : 3 A/D Samples 8

[Real Time Plot Parameters]

Time Window : 10 min
Iconize Real Time Display : False

Scale minimum : 0
Scale maximum : 1000000
Plot 1 type : No plot

Temperature Information
---- ----------- -----------

[GC Zone Temperatures]

Inj. A : 150 C Off
Inj. B : 0 C Off
Det. A : 175 C
Det. B : 250 C

[Oven Parameters]

Oven Equib Time : 0.10 min
Oven Max : 260 C
Oven : On
Cryo : On

[Oven Program]

Initial Temp. : 20 C
Initial Time : 2.00 min

Level	Rate (C/min)	Final Temp. (C)	Final Time (min)
	8.00	152	0.00
	30.00	220	3.23
3	0.00		

Next Run Time : 24.00 min

Injector Information
-------- -----------

Injection Source : Manual

[Purge Information]

Purge A/B	Init. Value	On Time	Off Time
A	Off	0.75	0.00
B	On	0.00	0.00

END OF ACQUISITION PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: D:\MS02\METHODS\MS28260J.M

Percent Report Settings

Sort By: Retention Time

Output Destination
    Screen: No
    Printer: No
    File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
1 75K	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
    Screen: No
    Printer: No
    File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination
    Screen: Yes
    Printer: No
    File: No

Generate Report During Run Method: No

TOPLEVEL PARAMETERS

Method Information For: D:\MS04\METHODS\524W2.M

Method Sections To Run:

( ) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
( ) Data Analysis
(X) Post-Run Cmd/Macro = macro "loctonet.mac",g

Method Comments:
EPA METHOD 8260/APP on 0.25 DB-624 column

END OF TOPLEVEL PARAMETERS

ACQUISITION PARAMETERS

General Information
------- -----------

et : GC
Tune File : BFB.U
Acquisition Mode : Scan

MS Information
-- -----------

Solvent Delay : 4.00 min

EM Absolute : False
EMV Offset : 105.9
Resulting Voltage : 2105.9

[Scan Parameters]

Low Mass : 35
High Mass : 260
Threshold : 500
Sampling # : 3 A/D Samples 8

[Real Time Plot Parameters]

me Window : 10 min
nize Real Time Display : False
Plot 1 type : Total ion

Scale maximum : 4000000
Plot 1 Sig= No plot

GC Inlet Information

[Inlet A Temperature Program Information]

Oven Track : Off
Inlet A Off

[Inlet B Temperature Program Information]

Oven Track : Off
Initial Temp. : 250 C
Initial Time : 480.00 min

Level	Rate (C/min)	Final Temp. (C)	Final Time (min)
1	0		

Total Program Time: 480.00 min

[Inlet A Pressure Program Information]

Constant Flow : Off
Initial Pres. : 10.0 psi
Initial Time : 480.00 min

el	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
	0		

Total Program Time: 480.00 min
Pressure Units : psi

[Inlet A Flow Settings]

Column length : 60.00 m
Column diameter : 0.250 mm
Gas : He
Vacuum compensation : Off
Pressure : 26.7 psi
Flow : 1.5 ml/min
Linear velocity : 26.8 cm/sec

[Inlet B Pressure Program Information]

Constant Flow : On 8.0 psi at 40 C
Pressure Units : psi

[Inlet B Flow Settings]

olumn length : 60.00 m
lumn diameter : 0.320 mm
Gas : He

Pressure : 8.3 psi
Flow : 1.5 ml/min
Linear velocity : 31.2 cm/sec
Split flow : 20 ml/min
Split ratio : 13.3

[Auxiliary Channel C Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	0		

Total Program Time: 480.00 min

[Auxiliary Channel D Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	0		

al Program Time: 480.00 min

[Auxiliary Channel E Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	0		

Total Program Time: 480.00 min

[Auxiliary Channel F Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

el	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	0		

Temperature Information

[GC Zone Temperatures]

Inj. A : 170 C Off
Inj. B : 250 C
Det. A : 170 C Off
Det. B : 250 C
Aux. : 280 C Off

[Oven Parameters]

Oven Equib Time : 0.20 min
Oven Max : 275 C
Oven : On
Cryo : Off
Ambient : 35 C
Cryo Blast : Off

[Oven Program]

Initial Temp. : 45 C
Initial Time : 2.00 min

Level	Rate (C/min)	Final Temp. (C)	Final Time (min)
	7.50	90	1.00
2	15.00	160	0.00
3	20.00	230	6.70

Next Run Time : 23.87 min

Injector Information

Injection Source : Manual

[Purge Information]

Purge A/B	Init. Value	On Time	Off Time
A	On	0.00	0.00
B	On	0.00	0.00

END OF ACQUISITION PARAMETERS

Method Information For: D:\MS08\METHODS\A_8260.M

Method Sections To Run:

( ) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
(X) Post-Run Cmd/Macro = macro "loctonet.mac",g

Method Comments:
EPA METHOD 8260 FOR SOILS

END OF TOPLEVEL PARAMETERS

ACQUISITION PARAMETERS

General Information

et	: GC
e File	: SOIL_BFB.U
Acquisition Mode	: Scan

MS Information

Solvent Delay	: 4.30 min
EM Absolute	: False
EMV Offset	: 0.0
Resulting Voltage	: 2235.3

[Scan Parameters]

Low Mass	: 35
High Mass	: 260
Threshold	: 500
Sampling #	: 3 A/D Samples 8

[Real Time Plot Parameters]

me Window	: 10 min
onize Real Time Display	: False
lot 1 type	: Total ion

Scale maximum : 1000000
Plot 1 type : No plot

GC Inlet Information

[Inlet A Temperature Program Information]

Oven Track : Off
Inlet A Off

[Inlet B Temperature Program Information]

Oven Track : Off
Initial Temp. : 250 C
Initial Time : 480.00 min

Level	Rate (C/min)	Final Temp. (C)	Final Time (min)
1	0		

Total Program Time: 480.00 min

[Inlet A Pressure Program Information]

Constant Flow : On 4.8 psi at 100 C
Pressure Units : psi

let A Flow Settings]

Column length : 60.00 m
Column diameter : 0.320 mm
Gas : He
Vacuum compensation : On
Pressure : 7.1 psi
Flow : 1.0 ml/min
Linear velocity : 26.2 cm/sec
Split flow : 25 ml/min
Split ratio : 25.0

[Inlet B Pressure Program Information]

Constant Flow : On 4.1 psi at 100 C
Pressure Units : psi

[Inlet B Flow Settings]

Column length : 60.00 m
Column diameter : 0.320 mm
Gas : He
Vacuum compensation : On
essure : 4.2 psi
ow : 0.8 ml/min
Linear velocity : 22.7 cm/sec

[Auxiliary Channel C Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	0		

Total Program Time: 480.00 min

[Auxiliary Channel D Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	0		

Total Program Time: 480.00 min

xiliary Channel E Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	0		

Total Program Time: 480.00 min

[Auxiliary Channel F Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	0		

Total Program Time: 480.00 min

GC Temperature Information
--------- -----------

[GC Zone Temperatures]

	A : 170 C	Off
Inj.	B : 250 C	
Det.	A : 170 C	Off
Det.	B : 250 C	
Aux.	: 280 C	

[Oven Parameters]

Oven Equib Time	: 0.00 min
Oven Max	: 225 C
Oven	: On
Cryo	: On
Ambient	: 35 C
Cryo Blast	: Off

[Oven Program]

Initial Temp. : 40 C
Initial Time : 1.50 min

Level	Rate (C/min)	Final Temp. (C)	Final Time (min)
1	10.00	200	0.00
2	30.00	220	5.83
3	0.00		

Next Run Time : 24.00 min

Injector Information
-------- -----------

Injection Source : Manual

[Purge Information]

Purge A/B	Init. Value	On Time	Off Time
A	On	0.00	0.00
B	On	0.00	0.00

END OF ACQUISITION PARAMETERS

DATA ANALYSIS PARAMETERS

Method Information For: D:\MS13\METHODS\8260W4.M

Method Sections To Run:

( ) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
(X) Post-Run Cmd/Macro = macro "loctonet.mac",g

Method Comments:

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: External Device
Injection Location: Front
Mass Spectrometer: Enabled

HP6890 GC METHOD

OVEN
Initial temp: 40 'C (On)
Initial time: 2.00 min
Maximum temp: 260 'C
Equilibration time: 0.00 min
Ramps:

#	Rate	Final temp	Final time
1	4.00	65	1.00
2	15.00	200	1.00
3	0.0(Off)		

Post temp: 0 'C
Post time: 0.00 min
Run time: 19.25 min

FRONT INLET (UNKNOWN)
Mode: Split
Initial temp: 200 'C (On)
Pressure: 14.26 psi (On)
Split ratio: 100:1
Split flow: 68.8 mL/min
Total flow: 72.8 mL/min
Gas saver: On
Saver flow: 20.0 mL/min
Saver time: 2.00 min

BACK INLET ()

COLUMN 1
Capillary Column
Model Number: J&W 1211324
db-624
Max temperature: 260 'C
Nominal length: 20.0 m
Nominal diameter: 180.00 um
Nominal film thickness: 1.00 um
Mode: constant flow
Initial flow: 0.7 mL/min
Nominal init pressure: 14.27 psi
Average velocity: 37 cm/sec
Inlet: Front Inlet
Outlet: (unspecified)
Outlet pressure: vacuum

COLUMN 2
(not installed)

FRONT DETECTOR (NO DET)

SIGNAL 1
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

BACK DETECTOR (NO DET)

SIGNAL 2
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
(No Detectors Installed)

COLUMN COMP 2
(No Detectors Installed)

THERMAL AUX 2
Use: MSD Transfer Line Heater
Description:
Initial temp: 250 'C (On)
Initial time: 0.00 min

#	Rate	Final temp	Final time
1	0.0	(Off)	

POST RUN
Post Time: 0.00 min

TIME TABLE

Time	Specifier	Parameter & Setpoint

7673 Injector

Front Injector:

Sample Washes	0	
Sample Pumps	0	
Injection Volume	1.0	microliters
Syringe Size	10.0	microliters
Nanoliter Adapter	Off	
PostInj Solvent A Washes	0	
PostInj Solvent B Washes	0	
Viscosity Delay	0	seconds
Plunger Speed	Fast	

Sample Washes	0
Sample Pumps	0
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
Nanoliter Adapter	Off
PostInj Solvent A Washes	0
PostInj Solvent B Washes	0
Viscosity Delay	0 seconds
Plunger Speed	Fast

MS ACQUISITION PARAMETERS

General Information
------- -----------

Tune File : BFB1.U
Acquistion Mode : Scan

MS Information
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Solvent Delay : 1.02 min

EM Absolute : False
EM Offset : 106
Resulting EM Voltage : 1258.8

[Scan Parameters]

Low Mass : 35.0
High Mass : 260.0
Threshold : 200
Sample # : 3 A/D Samples 8

[MSZones]

MS Quad : 150 C maximum 200 C
MS Source : 250 C maximum 250 C

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: J:\MS13\METHODS\082804_8260W.M

Sort By: Signal

Output Destination
Screen: No
Printer: Yes
File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
L:\Database\NIST98.L	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
Screen: No
Printer: Yes
File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination
Screen: No
Printer: Yes
File: No

Generate Report During Run Method: No

VOA MS13 EPA Method 8260B
Calibration Last Updated: Sun Aug 29 17:56:26 2004

erence Window: 100.00 Minutes

ATTACHMENT B

INITIAL CALIB ,ON CURVE

Date_____ Analysis: 8260/App.

Prepared By_____ Instrument:

Matrix: Water

Stock Solution #1_____ Analytes: Surrogate Init. Concentration: 100ppm

Stock Solution #2_____ Analytes: Low 8260/Ketones Init. Concentration: 5/10/20/100/200ppm

Stock Solution #3_____ Analytes: 8260 Init. Concentration: 50/100/200/1000/2000ppm

Stock Solution #4_____ Analytes: Ketones Init. Concentration: 2000ppm

Aliquot of Stock Solution #1 (µL)	Final Conc. of #1 (µg/L)	Aliquot of Stock Solution #2 (µL)	Final Conc. of #2 (µg/L)	Aliquot of Stock Solution #3 (µL)	Final Conc. of #3 (µg/L)	Aliquot of Stock Solution #4 (µL)	Final Conc. of #4 (µg/L)	Final Volume (mL)	Notes
1.5	3	2.0	0.2/.4/.8/4/8					50	Level ID: 1
2.0	4	3.0	0.3/.6/1.2/6/12					50	Level ID: 2
2.5	5	5.0	.5/1/2/10/20					50	Level ID: 3
3.0	6	12.5	1.25/2.5/5/25/50					50	Level ID: 4
4.0	8	20	2/4/8/40/80					50	Level ID: 5
4.5	9			5	5/10/20/100/200	2.5	100	50	Level ID: 6
5.0	10			10	10/20/40/200/400	5.0	200	50	Level ID: 7
10	20			20	20/40/80/400/800	10	400	50	Level ID: 8
20	40			40	40/80/160/800/1600	20	800	50	Level ID: 9
25	50			80	80/160/320/1600/3200	40	1600	50	Level ID: 10

8260 ICV: 10uL of 50/250ppm Accustd ICV ( ) + 50uL of 100ppm Acrolein ( ) +

5uL of 100ppm Dichlorofluoromethane ( ) + 5uL of 200ppm n-Octane/Tetrahydrofuran ( ) +

7.5uL of 200/2000ppm Appendix ICV ( ) + 5uL of 200/1000ppm Oxygenates ( ) to 50mL.

INITIAL CALIBRATION CURVE

Date_____ Analysis: 8260

Prepared By_____ Instrument:

Matrix: Soil

Stock Solution #1_____ Analytes: Surrogate Init. Concentration: 100ppm

Stock Solution #2_____ Analytes: Low 8260/Ketones Init. Concentration: 10/20/40/50/100ppm

Stock Solution #3_____ Analytes: 8260/Ketones Init. Concentration: 100/200/400/500/1000ppm

Aliquot of Stock Solution #1 (µL)	Final Conc. of #1 (µg/L)	Aliquot of Stock Solution #2 (µL)	Final Conc. of #2 (µg/L)	Aliquot of Stock Solution #3 (µL)	Final Conc. of #3 (µg/L)			Final Volume (mL)	Notes
2.5	5.0	5.0	1.0/2.0/4.0/5.0/10					50	Level ID: 1
5.0	10	10	2.0/4.0/8.0/10/20					50	Level ID: 2
10	20	25	5.0/10/20/25/50					50	Level ID: 3
15	30	50	10/20/40/50/100					50	Level ID: 4
20	40			10	20/40/80/100/200			50	Level ID: 5
25	50			25	50/100/200/250/500			50	Level ID: 6
30	60			50	100/200/400/500/1000			50	Level ID: 7
40	80			150	300/600/1200/1500/3000			50	Level ID: 8

8260 ICV: 50uL of 50/250ppm Accustd ICV ( ) + 50uL of 100ppm Acrolein ( ) +

12.5uL of 200/1000ppm Oxygenates ( ) to 50mL.

ATTACHMENT C

VOLATILE ORGANICS ANALYSES						
Method	Prep Method	Matrix	Analyte	LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
8260B	5030A/5035	Soil-low	1,1,1,2-Tetrachloroethane	79-113	19-135	40
8260B	5030A/5035	Soil-low	1,1,1-Trichloroethane (TCA)	77-126	37-136	40
8260B	5030A/5035	Soil-low	1,1,2,2-Tetrachloroethane	69-113	12-150	40
8260B	5030A/5035	Soil-low	1,1,2-Trichloroethane	77-111	30-134	40
8260B	5030A/5035	Soil-low	1,1-Dichloroethane	76-119	41-132	40
8260B	5030A/5035	Soil-low	1,1-Dichloroethene	80-134	40-148	40
8260B	5030A/5035	Soil-low	1,1-Dichloropropene	80-125	36-139	40
8260B	5030A/5035	Soil-low	1,2,3-Trichlorobenzene	77-125	10-126	40
8260B	5030A/5035	Soil-low	1,2,3-Trichloropropane	74-114	12-162	40
8260B	5030A/5035	Soil-low	1,2,4-Trichlorobenzene	78-128	10-130	
8260B	5030A/5035	Soil-low	1,2,4-Trimethylbenzene	76-117	10-144	40
8260B	5030A/5035	Soil-low	1,2-Dibromo-3-chloropropane	71-112	10-147	40
8260B	5030A/5035	Soil-low	1,2-Dibromoethane (EDB)	79-111	29-134	40
8260B	5030A/5035	Soil-low	1,2-Dichlorobenzene	76-107	10-130	40
8260B	5030A/5035	Soil-low	1,2-Dichloroethane (EDC)	72-124	38-131	40
8260B	5030A/5035	Soil-low	1,2-Dichloropropane	79-118	40-130	40
8260B	5035/5030B	Soil-low	1,3,5-Trichlorobenzene	70-130	70-130	40
8260B	5030A/5035	Soil-low	1,3,5-Trimethylbenzene	75-116	10-158	40
8260B	5030A/5035	Soil-low	1,3-Dichlorobenzene	76-108	10-135	40
8260B	5030A/5035	Soil-low	1,3-Dichloropropane	77-112	34-136	40
8260B	5030A/5035	Soil-low	1,4-Dichlorobenzene	74-108	10-129	40
8260B	5030A/5035	Soil-low	1,4-Dioxane	10-172	70-130	40
8260B	5030A/5035	Soil-low	1-Chlorohexane	81-123	70-130	40
8260B	5030A/5035	Soil-low	2,2-Dichloropropane	78-131	40-139	40
8260B	5030A/5035	Soil-low	2-Butanone (MEK)	67-111	23-135	40
8260B	5030A/5035	Soil-low	2-Chloroethyl Vinyl Ether	64-151	13-143	40
8260B	5030A/5035	Soil-low	2-Chlorotoluene	75-116	10-146	40
8260B	5030A/5035	Soil-low	2-Hexanone	55-122	14-145	40
8260B	5030A/5035	Soil-low	2-Nitropropane	47-125	70-130	40
8260B	5030A/5035	Soil-low	3-Chloro-1-propene	49-109	70-130	40
8260B	5030A/5035	Soil-low	4-Chlorotoluene	72-114	10-142	40
8260B	5030A/5035	Soil-low	4-Isopropyltoluene	73-114	10-136	40
8260B	5030A/5035	Soil-low	4-Methyl-2-pentanone (MIBK)	67-120	23-147	40
8260B	5030A/5035	Soil-low	Acetone	47-110	15-125	40
8260B	5030A/5035	Soil-low	Acetonitrile	49-121	70-130	40
8260B	5030A/5035	Soil-low	Acrolein	10-188	10-166	40
8260B	5030A/5035	Soil-low	Acrylonitrile	70-130	43-173	40
8260B	5030A/5035	Soil-low	Benzene	75-126	38-132	40

VOLATILE ORGANICS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
8260B	5030A/5035	Soil-low	Bromobenzene	76-109	13-142	40
8260B	5030A/5035	Soil-low	Bromochloromethane	79-119	39-133	40
8260B	5030A/5035	Soil-low	Bromodichloromethane	82-127	26-142	40
8260B	5030A/5035	Soil-low	Bromoform	81-121	10-145	40
8260B	5030A/5035	Soil-low	Bromomethane	54-166	16-173	40
8260B	5030A/5035	Soil-low	Carbon Disulfide	69-138	24-145	40
8260B	5030A/5035	Soil-low	Carbon Tetrachloride	77-133	17-145	40
8260B	5030A/5035	Soil-low	Chlorobenzene	78-106	19-129	40
8260B	5030A/5035	Soil-low	Chloroethane	53-135	28-148	40
8260B	5030A/5035	Soil-low	Chloroform	78-117	41-128	40
8260B	5030A/5035	Soil-low	Chloromethane	50-146	29-145	40
8260B	5030A/5035	Soil-low	Chloroprene	47-129	70-130	40
8260B	5030A/5035	Soil-low	cis-1,2-Dichloroethene	84-124	37-141	40
8260B	5030A/5035	Soil-low	cis-1,3-Dichloropropene	84-121	26-136	40
8260B	5030A/5035	Soil-low	cis-1,4-Dichloro-2-butene	63-118	70-130	40
8260B	5030A/5035	Soil-low	Cyclohexane	70-130	70-130	40
8260B	5030A/5035	Soil-low	Dibromochloromethane	82-116	18-141	40
8260B	5030A/5035	Soil-low	Dibromomethane	79-118	37-133	40
8260B	5030A/5035	Soil-low	Dichlorodifluoromethane	34-161	23-156	40
8260B	5030A/5035	Soil-low	Dichlorofluoromethane (CFC 21)	30-167	70-130	40
8260B	5030A/5035	Soil-low	Diisopropyl Ether	69-130	70-130	40
8260B	5030A/5035	Soil-low	Ethyl Acetate	51-116	70-130	40
8260B	5030A/5035	Soil-low	Ethyl Acrylate	70-130	70-130	40
8260B	5030A/5035	Soil-low	Ethyl Methacrylate	62-115	70-130	40
8260B	5030A/5035	Soil-low	Ethylbenzene	79-111	18-137	40
8260B	5030A/5035	Soil-low	Hexachlorobutadiene	77-129	10-126	40
8260B	5030A/5035	Soil-low	Iodomethane	54-139	13-150	40
8260B	5030A/5035	Soil-low	Isobutanol	56-112	70-130	40
8260B	5030A/5035	Soil-low	Isopropylbenzene	73-104	10-124	40
8260B	5030A/5035	Soil-low	m,p-Xylenes	80-116	13-139	40
8260B	5030A/5035	Soil-low	Methacrylonitrile	59-110	70-130	40
8260B	5030A/5035	Soil-low	Methyl Acetate	70-130	70-130	40
8260B	5030A/5035	Soil-low	Methyl Methacrylate	57-107	70-130	40
8260B	5030A/5035	Soil-low	Methyl tert-Butyl Ether	76-116	31-142	40
8260B	5030A/5035	Soil-low	Methylcyclohexane	70-130	70-130	40
8260B	5030A/5035	Soil-low	Methylene Chloride	47-169	29-150	40
8260B	5030A/5035	Soil-low	n-Butylbenzene	75-122	10-139	40
8260B	5030A/5035	Soil-low	n-Hexane	70-130	70-130	40

VOLATILE ORGANICS ANALYSES							
Method	**Prep Method**	**Matrix**	**Analyte**		**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
8260B	5030A/5035	Soil-low	n-Propylbenzene		76-122	10-152	40
8260B	5030A/5035	Soil-low	Naphthalene		70-124	10-135	40
8260B	5030A/5035	Soil-low	o-Xylene		79-113	10-149	40
8260B	5030A/5035	Soil-low	Propionitrile		53-114	70-130	40
8260B	5030A/5035	Soil-low	sec-Butylbenzene		81-126	10-151	40
8260B	5030A/5035	Soil-low	Styrene		82-118	10-144	40
8260B	5030A/5035	Soil-low	tert-Amyl Methyl Ether		82-127	70-130	40
8260B	5030A/5035	Soil-low	tert-Butyl Alcohol		75-130	70-130	40
8260B	5030A/5035	Soil-low	tert-Butyl Ethyl Ether		77-124	70-130	40
8260B	5030A/5035	Soil-low	tert-Butylbenzene		75-117	10-144	40
8260B	5030A/5035	Soil-low	Tetrachloroethene (PCE)		81-113	13-142	40
8260B	5030A/5035	Soil-low	Toluene		77-115	26-133	40
8260B	5030A/5035	Soil-low	trans-1,2-Dichloroethene		80-118	36-135	40
8260B	5030A/5035	Soil-low	trans-1,3-Dichloropropene		74-109	20-129	40
8260B	5030A/5035	Soil-low	trans-1,4-Dichloro-2-butene		37-221	10-193	40
8260B	5030A/5035	Soil-low	Trichloroethene (TCE)		81-119	32-135	40
8260B	5030A/5035	Soil-low	Trichlorofluoromethane		57-129	28-135	40
8260B	5030A/5035	Soil-low	Trichlorotrifluoroethane		68-131	19-141	40
8260B	5030A/5035	Soil-low	Vinyl Acetate		10-159	10-143	40
8260B	5030A/5035	Soil-low	Vinyl Chloride		58-136	33-146	40
8260B	5030A/5035	Soil-low	1,2-Dichloroethane-D4 (Surr.)	60-120	NA	NA	NA
8260B	5030A/5035	Soil-low	4-Bromofluorobenzene (Surr.)	58-117	NA	NA	NA
8260B	5030A/5035	Soil-low	Dibromofluoromethane (Surr.)	61-116	NA	NA	NA
8260B	5030A/5035	Soil-low	Toluene-D8 (Surr.)	63-116	NA	NA	NA
8260B	5035/5030B	Soil-mid	1,1,1,2-Tetrachloroethane		70-122	63-122	40
8260B	5035/5030B	Soil-mid	1,1,1-Trichloroethane (TCA)		69-126	62-126	40
8260B	5035/5030B	Soil-mid	1,1,2,2-Tetrachloroethane		71-126	53-141	40
8260B	5035/5030B	Soil-mid	1,1,2-Trichloroethane		79-120	72-121	40
8260B	5035/5030B	Soil-mid	1,1-Dichloroethane		79-120	72-118	40
8260B	5035/5030B	Soil-mid	1,1-Dichloroethene		79-132	68-132	40
8260B	5035/5030B	Soil-mid	1,1-Dichloropropene		75-122	71-121	40
8260B	5035/5030B	Soil-mid	1,2,3-Trichlorobenzene		65-117	64-119	40
8260B	5035/5030B	Soil-mid	1,2,3-Trichloropropane		67-132	54-151	40
8260B	5035/5030B	Soil-mid	1,2,4-Trichlorobenzene		68-110	65-116	40
8260B	5035/5030B	Soil-mid	1,2,4-Trimethylbenzene		80-121	67-130	40
8260B	5035/5030B	Soil-mid	1,2-Dibromo-3-chloropropane		58-119	52-134	
8260B	5035/5030B	Soil-mid	1,2-Dibromoethane (EDB)		74-121	72-119	40
8260B	5035/5030B	Soil-mid	1,2-Dichlorobenzene		80-112	77-109	40

VOLATILE ORGANICS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
8260B	5035/5030B	Soil-mid	1,2-Dichloroethane (EDC)	67-129	66-123	40
8260B	5035/5030B	Soil-mid	1,2-Dichloropropane	81-117	74-117	40
8260B	5035/5030B	Soil-mid	1,3,5-Trichlorobenzene	59-114	70-130	40
8260B	5035/5030B	Soil-mid	1,3,5-Trimethylbenzene	79-119	72-121	40
8260B	5035/5030B	Soil-mid	1,3-Dichlorobenzene	80-112	75-111	40
8260B	5035/5030B	Soil-mid	1,3-Dichloropropane	80-119	76-116	40
8260B	5035/5030B	Soil-mid	1,4-Dichlorobenzene	80-113	73-111	40
8260B	5035/5030B	Soil-mid	1,4-Dioxane	35-178	70-130	40
8260B	5035/5030B	Soil-mid	1-Chlorohexane	75-118	70-130	40
8260B	5035/5030B	Soil-mid	2,2-Dichloropropane	42-132	38-143	40
8260B	5035/5030B	Soil-mid	2-Butanone (MEK)	70-117	69-124	40
8260B	5035/5030B	Soil-mid	2-Chloroethyl Vinyl Ether	33-155	40-163	40
8260B	5035/5030B	Soil-mid	2-Chlorotoluene	83-119	72-127	40
8260B	5035/5030B	Soil-mid	2-Hexanone	66-118	56-138	40
8260B	5035/5030B	Soil-mid	2-Nitropropane	10-99	70-130	40
8260B	5035/5030B	Soil-mid	3-Chloro-1-propene	40-85	70-130	40
8260B	5035/5030B	Soil-mid	4-Chlorotoluene	81-117	70-125	40
8260B	5035/5030B	Soil-mid	4-Isopropyltoluene	71-114	63-123	40
8260B	5035/5030B	Soil-mid	4-Methyl-2-pentanone (MIBK)	68-115	62-126	40
8260B	5035/5030B	Soil-mid	Acetone	59-114	59-118	40
8260B	5035/5030B	Soil-mid	Acetonitrile	70-130	70-130	40
8260B	5035/5030B	Soil-mid	Acrolein	10-281	37-253	40
8260B	5035/5030B	Soil-mid	Acrylonitrile	10-147	10-138	40
8260B	5035/5030B	Soil-mid	Benzene	82-115	71-117	40
8260B	5035/5030B	Soil-mid	Bromobenzene	78-121	75-117	40
8260B	5035/5030B	Soil-mid	Bromochloromethane	74-129	72-128	40
8260B	5035/5030B	Soil-mid	Bromodichloromethane	75-121	70-121	40
8260B	5035/5030B	Soil-mid	Bromoform	67-131	59-132	40
8260B	5035/5030B	Soil-mid	Bromomethane	23-121	10-126	40
8260B	5035/5030B	Soil-mid	Carbon Disulfide	73-125	64-129	40
8260B	5035/5030B	Soil-mid	Carbon Tetrachloride	60-129	56-128	40
8260B	5035/5030B	Soil-mid	Chlorobenzene	82-116	74-117	40
8260B	5035/5030B	Soil-mid	Chloroethane	57-125	58-130	40
8260B	5035/5030B	Soil-mid	Chloroform	66-124	65-118	40
8260B	5035/5030B	Soil-mid	Chloromethane	49-124	48-134	40
8260B	5035/5030B	Soil-mid	Chloroprene	23-101	70-130	40
8260B	5035/5030B	Soil-mid	cis-1,2-Dichloroethene	86-125	73-129	40
8260B	5035/5030B	Soil-mid	cis-1,3-Dichloropropene	73-121	68-124	40

VOLATILE ORGANICS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
8260B	5035/5030B	Soil-mid	cis-1,4-Dichloro-2-butene	36-158	70-130	40
8260B	5035/5030B	Soil-mid	Cyclohexane	70-130	70-130	40
8260B	5035/5030B	Soil-mid	Dibromochloromethane	66-131	62-126	40
8260B	5035/5030B	Soil-mid	Dibromomethane	73-127	69-123	40
8260B	5035/5030B	Soil-mid	Dichlorodifluoromethane	10-168	21-164	40
8260B	5035/5030B	Soil-mid	Dichlorofluoromethane (CFC 21)	76-137	70-130	40
8260B	5035/5030B	Soil-mid	Diisopropyl Ether	78-121	70-130	40
8260B	5035/5030B	Soil-mid	Ethyl Acetate	28-85	70-130	40
8260B	5035/5030B	Soil-mid	Ethyl Ether	33-148	70-130	40
8260B	5035/5030B	Soil-mid	Ethyl Methacrylate	29-98	70-130	40
8260B	5035/5030B	Soil-mid	Ethylbenzene	80-121	72-124	40
8260B	5035/5030B	Soil-mid	Hexachlorobutadiene	57-129	50-136	40
8260B	5035/5030B	Soil-mid	Iodomethane	20-190	55-141	40
8260B	5035/5030B	Soil-mid	Isobutanol	18-171	70-130	40
8260B	5035/5030B	Soil-mid	Isopropylbenzene	75-108	53-124	40
8260B	5035/5030B	Soil-mid	m,p-Xylenes	82-118	71-127	40
8260B	5035/5030B	Soil-mid	Methacrylonitrile	39-84	70-130	40
8260B	5035/5030B	Soil-mid	Methyl Acetate	70-130	70-130	40
8260B	5035/5030B	Soil-mid	Methyl Methacrylate	33-91	70-130	40
8260B	5035/5030B	Soil-mid	Methyl tert-Butyl Ether	65-120	68-119	40
8260B	5035/5030B	Soil-mid	Methylcyclohexane	70-130	70-130	40
8260B	5035/5030B	Soil-mid	Methylene Chloride	69-125	69-117	40
8260B	5035/5030B	Soil-mid	n-Butylbenzene	65-120	55-135	40
8260B	5035/5030B	Soil-mid	n-Hexane	72-125	70-130	40
8260B	5035/5030B	Soil-mid	n-Propylbenzene	80-119	73-122	40
8260B	5035/5030B	Soil-mid	Naphthalene	71-125	48-161	40
8260B	5035/5030B	Soil-mid	o-Xylene	83-118	74-124	40
8260B	5035/5030B	Soil-mid	Propionitrile	36-89	70-130	40
8260B	5035/5030B	Soil-mid	sec-Butylbenzene	81-130	71-134	40
8260B	5035/5030B	Soil-mid	Styrene	89-122	77-128	40
8260B	5035/5030B	Soil-mid	tert-Amyl Methyl Ether	78-122	70-130	40
8260B	5035/5030B	Soil-mid	tert-Butyl Alcohol	26-165	70-130	40
8260B	5035/5030B	Soil-mid	tert-Butyl Ethyl Ether	60-127	70-130	40
8260B	5035/5030B	Soil-mid	tert-Butylbenzene	77-120	70-125	40
8260B	5035/5030B	Soil-mid	Tetrachloroethene (PCE)	71-128	58-129	40
8260B	5035/5030B	Soil-mid	Toluene	79-117	65-120	40
8260B	5035/5030B	Soil-mid	trans-1,2-Dichloroethene	79-118	70-118	40
8260B	5035/5030B	Soil-mid	trans-1,3-Dichloropropene	58-120	52-127	40

VOLATILE ORGANICS ANALYSES							
Method	**Prep Method**	**Matrix**	**Analyte**		**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
8260B	5035/5030B	Soil-mid	trans-1,4-Dichloro-2-butene		57-149	63-152	40
8260B	5035/5030B	Soil-mid	Trichloroethene (TCE)		75-127	63-130	40
8260B	5035/5030B	Soil-mid	Trichlorofluoromethane		57-126	53-122	40
8260B	5035/5030B	Soil-mid	Trichlorotrifluoroethane		64-128	62-131	40
8260B	5035/5030B	Soil-mid	Vinyl Acetate		10-140	10-137	40
8260B	5035/5030B	Soil-mid	Vinyl Chloride		58-129	57-141	40
8260B	5035/5030B	Soil-mid	1,2-Dichloroethane-D4[g]	66-119	NA	NA	NA
8260B	5035/5030B	Soil-mid	4-Bromofluorobenzene[g]	58-135	NA	NA	NA
8260B	5035/5030B	Soil-mid	Dibromofluoromethane[g]	69-118	NA	NA	NA
8260B	5035/5030B	Soil-mid	Toluene-D8[g]	77-123	NA	NA	NA
8260B	5030B	Water	1,1,1,2-Tetrachloroethane		71-118	66-123	30
8260B	5030B	Water	1,1,1-Trichloroethane (TCA)		65-126	63-137	30
8260B	5030B	Water	1,1,2,2-Tetrachloroethane		63-126	64-127	30
8260B	5030B	Water	1,1,2-Trichloroethane		73-122	68-125	30
8260B	5030B	Water	1,1-Dichloroethane		72-122	67-130	30
8260B	5030B	Water	1,1-Dichloroethene		70-136	67-147	30
8260B	5030B	Water	1,1-Dichloropropene		71-119	66-132	30
8260B	5030B	Water	1,2,3-Trichlorobenzene		64-120	61-124	30
8260B	5030B	Water	1,2,3-Trichloropropane		67-123	65-122	30
8260B	5030B	Water	1,2,4-Trichlorobenzene		66-115	60-122	30
8260B	5030B	Water	1,2,4-Trimethylbenzene		72-121	67-127	30
8260B	5030B	Water	1,2-Dibromo-3-chloropropane		49-124	46-134	30
8260B	5030B	Water	1,2-Dibromoethane (EDB)		71-120	66-121	30
8260B	5030B	Water	1,2-Dichlorobenzene		75-110	67-116	30
8260B	5030B	Water	1,2-Dichloroethane (EDC)		69-125	65-126	30
8260B	5030B	Water	1,2-Dichloropropane		73-123	70-127	30
8260B	5030B	Water	1,3,5-Trichlorobenzene		46-133	60-134	30
8260B	5030B	Water	1,3,5-Trimethylbenzene		70-120	63-128	30
8260B	5030B	Water	1,3-Dichlorobenzene		76-110	68-117	30
8260B	5030B	Water	1,3-Dichloropropane		74-120	68-122	30
8260B	5030B	Water	1,4-Dichlorobenzene		74-112	68-115	30
8260B	5030B	Water	1,4-Dioxane		19-191	37-173	30
8260B	5030B	Water	1-Chlorohexane		65-121	54-139	30
8260B	5030B	Water	2,2-Dichloropropane		48-133	43-147	30
8260B	5030B	Water	2-Butanone (MEK)		56-137	54-136	30
8260B	5030B	Water	2-Chloroethyl Vinyl Ether		10-192	10-170	30
8260B	5030B	Water	2-Chlorotoluene		72-120	63-131	30
8260B	5030B	Water	2-Hexanone		44-135	44-135	30

VOLATILE ORGANICS ANALYSES						
Method	**Prep Method**	**Matrix**	**Analyte**	**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
8260B	5030B	Water	2-Nitropropane	10-169	43-138	30
8260B	5030B	Water	3-Chloro-1-propene	42-154	65-154	30
8260B	5030B	Water	4-Chlorotoluene	70-118	65-123	30
8260B	5030B	Water	4-Isopropyltoluene	67-115	63-122	30
8260B	5030B	Water	4-Methyl-2-pentanone (MIBK)	57-129	55-131	30
8260B	5030B	Water	Acetone	53-129	50-126	30
8260B	5030B	Water	Acetonitrile	58-143	63-150	30
8260B	5030B	Water	Acrolein	10-214	10-209	30
8260B	5030B	Water	Acrylonitrile	48-144	67-137	30
8260B	5030B	Water	Benzene	74-118	69-126	30
8260B	5030B	Water	Bromobenzene	76-111	69-117	30
8260B	5030B	Water	Bromochloromethane	72-123	69-125	30
8260B	5030B	Water	Bromodichloromethane	72-127	69-131	30
8260B	5030B	Water	Bromoform	57-135	54-136	30
8260B	5030B	Water	Bromomethane	24-144	24-148	30
8260B	5030B	Water	Carbon Disulfide	64-129	63-139	30
8260B	5030B	Water	Carbon Tetrachloride	58-133	56-143	30
8260B	5030B	Water	Chlorobenzene	74-115	68-120	30
8260B	5030B	Water	Chloroethane	60-128	57-135	30
8260B	5030B	Water	Chloroform	71-118	65-125	30
8260B	5030B	Water	Chloromethane	45-135	43-142	30
8260B	5030B	Water	Chloroprene	34-165	49-155	30
8260B	5030B	Water	cis-1,2-Dichloroethene	76-125	68-134	30
8260B	5030B	Water	cis-1,3-Dichloropropene	71-125	66-129	30
8260B	5030B	Water	cis-1,4-Dichloro-2-butene	10-176	34-175	30
8260B	5030B	Water	Dibromochloromethane	67-124	61-127	30
8260B	5030B	Water	Dibromomethane	71-124	69-124	30
8260B	5030B	Water	Dichlorodifluoromethane	21-156	16-168	30
8260B	5030B	Water	Dichlorofluoromethane (CFC 21)	68-139	70-130	30
8260B	5030B	Water	Diisopropyl Ether	70-129	70-128	30
8260B	5030B	Water	Ethyl Acetate	28-157	63-133	30
8260B	5030B	Water	Ethyl Ether	10-170	61-121	30
8260B	5030B	Water	Ethyl Methacrylate	30-166	71-161	30
8260B	5030B	Water	Ethylbenzene	71-118	65-126	30
8260B	5030B	Water	Hexachlorobutadiene	61-124	52-134	30
8260B	5030B	Water	Iodomethane	37-158	53-160	30
8260B	5030B	Water	Isobutanol	10-177	27-182	30
8260B	5030B	Water	Isopropylbenzene	65-110	59-118	30

VOLATILE ORGANICS ANALYSES							
Method	**Prep Method**	**Matrix**	**Analyte**		**LCS Accuracy (% Rec.)**	**Matrix Spike (% Rec.)**	**Precision (RPD)**
8260B	5030B	Water	m,p-Xylenes		73-119	63-130	30
8260B	5030B	Water	Methacrylonitrile		38-152	60-148	30
8260B	5030B	Water	Methyl Methacrylate		29-163	54-154	30
8260B	5030B	Water	Methyl tert-Butyl Ether		64-125	60-132	30
8260B	5030B	Water	Methylene Chloride		64-137	62-136	30
8260B	5030B	Water	n-Butylbenzene		62-123	59-130	30
8260B	5030B	Water	n-Hexane		44-150	58-154	30
8260B	5030B	Water	n-Octane		26-173	53-146	30
8260B	5030B	Water	n-Propylbenzene		69-122	65-129	30
8260B	5030B	Water	Naphthalene		58-132	61-137	30
8260B	5030B	Water	o-Xylene		74-120	65-130	30
8260B	5030B	Water	Propionitrile		38-152	58-141	30
8260B	5030B	Water	sec-Butylbenzene		73-130	68-137	30
8260B	5030B	Water	Styrene		75-123	66-130	30
8260B	5030B	Water	tert-Amyl Methyl Ether		68-132	57-139	30
8260B	5030B	Water	tert-Butyl Alcohol		24-166	53-149	30
8260B	5030B	Water	tert-Butyl Ethyl Ether		64-128	70-123	30
8260B	5030B	Water	tert-Butyl Formate		10-175	10-147	30
8260B	5030B	Water	tert-Butylbenzene		72-118	67-126	30
8260B	5030B	Water	Tetrachloroethene (PCE)		65-121	60-128	30
8260B	5030B	Water	Tetrahydrofuran		10-178	58-122	30
8260B	5030B	Water	Toluene		74-117	66-128	30
8260B	5030B	Water	trans-1,2-Dichloroethene		70-121	69-128	30
8260B	5030B	Water	trans-1,3-Dichloropropene		56-121	53-123	30
8260B	5030B	Water	trans-1,4-Dichloro-2-butene		28-174	57-172	30
8260B	5030B	Water	Trichloroethene (TCE)		71-122	56-137	30
8260B	5030B	Water	Trichlorofluoromethane		54-129	52-138	30
8260B	5030B	Water	Trichlorotrifluoroethane		59-130	57-141	30
8260B	5030B	Water	Vinyl Acetate		10-193	29-143	30
8260B	5030B	Water	Vinyl Chloride		59-135	52-151	30
8260B	5030B	Water	1,2-Dichloroethane-D4 (Surr.)	62-121	NA	NA	NA
8260B	5030B	Water	4-Bromofluorobenzene (Surr.)	75-117	NA	NA	NA
8260B	5030B	Water	Dibromofluoromethane (Surr.)	75-120	NA	NA	NA
8260B	5030B	Water	Toluene-D8 (Surr.)	80-128	NA	NA	NA

C-6

STANDARD OPERATING PROCEDURE

for

PURGE and TRAP/EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES, CLOSED SYSTEM

SOP No.: VOC-5035

Revision: 7

February 1, 2008

Approved By:

Supervisor	2/1/8 Date
QA Manager	2-4-08 Date
Laboratory Manager	2/4/08 Date

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Annual review of this SOP has been performed and the SOP still reflects current practice.
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Initials: _____ Date: _____

PURGE-AND-TRAP/ EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES, CLOSED SYSTEM

1. SCOPE AND APPLICATION

1.1. This procedure describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste) using EPA Method 5035 and 5035A. While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260.

1.2. The low concentration procedure is generally applicable to soils and other solid samples with VOC concentrations in the range of 0.5 to 200 µg/kg, but may be dependent on the determinative method, matrix, and compound . Procedures are included for preparing high concentration samples (those containing VOC levels of >200 µg/kg) for purging using Method 5030.

1.3. Procedures for handling multi-increment sampling are included in the SOP. Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent. These samples are also purged using Method 5030.

1.4. Methods 5035 and 5035A can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

1.5. Methods 5035 and 5035A, in conjunction with Method 8015 (GC/FID), may be used for the analysis of the aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons, e.g., gasoline. For the aromatic fraction (BTEX), use Method 8021 (GC/PID) in conjunction with this procedure. A total determinative analysis of gasoline fractions may be obtained using Method 8021 in series with Method 8015.

1.6. As with any preparative method for volatiles, samples should be screened to avoid contamination of the purge-and-trap system by samples that contain very high concentrations of purgeable material above the calibration range of the low concentration method. In addition, because the sealed sample container cannot be opened to remove a sample aliquot without compromising the integrity of the sample, multiple sample aliquots should be collected to allow for screening and reanalysis.

1.7. The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030.

1.8. This procedure is not applicable to samples that contain oily materials that are not soluble in water-miscible solvents.

2. SUMMARY OF METHOD

2.1. The low soil method utilizes a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. The VOCs are determined by collecting an approximate 5g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap that already contains a stirring bar and a sodium bisulfate preservative solution. The vial is sealed. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free reagent water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. Alternatively, soil samples may be collected in Encore sampling devices and stored or prepared at the lab prior to analysis. The sample vial is heated to 40°C and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2. The high concentration soil procedure is generally applicable to soils and other solid samples with VOC concentrations greater than 400 µg/kg. The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 400 µg/kg) of VOCs which may overload either the trapping material or exceed the working range of the instrument. In such instances, this procedure describes two sample collection options and the corresponding sample purging procedures.

2.2.1. The first option is to collect an approximately 5g sample in a pre-weighed vial, with a septum-sealed screw-cap, that contains 5 mL of methanol. To analyze, an aliquot of the solvent is removed from the vial, diluted into reagent water, purged, and analyzed.

2.2.2. The second option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is extracted with methanol. An aliquot of the methanol solution is added to blank water in a graduated cylinder and brought to 50mL. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method.

2.3. Some projects may employ multi-increment (MI) sampling in the field. Some studies have shown that MI sampling, using 30+ sample increments within a decision unit (a defined field sampling area) may provide a more representative view of contaminant concentrations than

traditional discrete sampling approaches. References listed provide additional background on MI sampling. When this approach is taken it is important that laboratory procedures are consistent with field procedures when taking samples.

2.4. The waste procedure is generally applicable to samples with VOC concentrations > 200 µg/kg that can be diluted in a water-miscible solvent. This procedure is generally appropriate for samples that are comprised of oils or samples that have significant amounts of oil present, when they are soluble in methanol. After demonstrating that a test aliquot of the sample is soluble in methanol, a separate aliquot of the sample is spiked with surrogates and diluted in the appropriate solvent. An aliquot of the solution is added to blank water in a graduated cylinder and brought to 50 mL, taking care to ensure that a floating oil layer is not present. Internal standards (if applicable) are added to the solution which is then purged using Method 5030 and analyzed.

3. DEFINITIONS

3.1. Field preservation - The addition of sample preservative material or reagent to the sample at the time of sampling to aid in maintaining sample integrity and preventing biodegradation or chemical degradation of target compounds. The sample container may or may not be shipped to sampling staff with the preservative added prior to shipment.

3.2. Closed system purge and trap - A purge and trap system utilizing a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible.

3.3. Trip blank - A sample vial containing organic-free reagent water and carried through sampling and handling protocols to serve as a check on contamination by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage.

3.4. Multi-increment (MI) sample – A field sample consisting of multiple bulk containers from one decision unit (defined in a MI sampling plan) submitted to the lab for subsampling into a representative sample for analysis.

4. INTERFERENCES

4.1. Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap can account for contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation, resulting in interferences or false positives in the determinative step.

4.2. A trip blank should be included in sample shipments as a check on contamination of the sample vial during shipment and storage. Trip blanks are prepared and labeled as outlined in the SOP for Bottle Order Preparation and Shipping (SMO-BORD).

4.3. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free reagent water is not necessary.

4.4. The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken to minimize the presence of methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels may result.

4.5. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing.

4.6. Laboratory workers' clothing previously exposed to methylene chloride during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

6. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1. Refer to the introductory material for organic analytes in SW-846 for general sample collection information. EPA Methods 5035 and 5035A contain several options for collection and preservation of samples.

NOTE: At the time of this revision, several states and EPA regional offices have established guidelines for holding times and preservation of samples collected for low-level analysis using

the Encore®, or similar, sampler. The laboratory must ensure that these state or regional EPA protocols are followed.

6.2. Projects for MI samples will include additional instructions not found in the analytical SOP. The analyst should consult with the Project Chemist, or refer to the Project Chemist's instructions, prior to working with these samples.

6.3. Preparation of sample vials - The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and waste samples. Sample vials are prepared by the laboratory and provided to the client. Gloves should be worn during the preparation steps.

6.4. Not every option allowed under EPA SW-846 is described in this procedure. The following techniques listed are the laboratory's preferred and default procedures. Other sample preservation techniques, as described in Method 5035, 5035A, or a project QAPP may be required on a project-specific basis.

6.5. Low concentration soil samples - Soil samples to be analyzed by the closed-system purge-and-trap equipment. The low concentration portion of this procedure includes filling and weighing of vials in the field, which are never opened during the analytical process. As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

6.5.1. Collect the sample according to the procedures outlined in the sampling plan. Care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Always wear gloves whenever handling the tared sample vials. Preferred low concentration sample collection and preservation options are as follows:

Collection	Preservation	Holding Time	Comment
Encore®, or similar, coring device	Cool the coring device to 4 ± 2°C.	48 hours	
Encore®, or similar, coring device	Sample is extruded into an empty vial and frozen on-site to -7 to -20°C.	14 days	
Encore®, or similar, coring device	Sample is extruded into a vial containing reagent water, cooled to 4 ±2°C (no more than 48 hours), then frozen to -7 to -20°C.	14 days	Analysis must be completed within 48 hours if samples are not frozen prior to the expiration of the 48 hour period.
Bulk soil sampling jar.	No headspace, cool to 4 ± 2°C.	14 days	For projects using 5030A type sampling.

6.5.2. Preparation of VOA vials used in the collection of low concentration samples

6.5.2.1. Add a clean magnetic stirring bar to each clean vial.

6.5.2.2. Where samples will be extruded into water, add 5 mL of organic-free reagent water to each vial.

6.5.2.3. Seal the vial with the screw-cap and septum seal.

6.5.2.4. Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label.

6.5.2.5. Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

6.5.3. Sample collection (Encore®, or similar)

6.5.3.1. Follow the procedures provided with the sampling device. Collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere, generally within a few minutes at most.

6.5.3.2. Carefully wipe the exterior of the sample collection device with a clean cloth or towel. Extrude the sample into the appropriate vial, or cap the sampler and return it to the sampler bag. For samples extruded into vials, the sampler must then weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed. Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

6.5.3.3. Collect at least two replicate (preferably three) samples. This will allow the laboratory an additional sample for reanalysis. At least one additional aliquot of sample must be collected for screening and/or high concentration analysis (if necessary). The third aliquot may be collected in a 60-mL glass vial or a third 40-mL soil sample vial. To determine dry weight, a sample not containing the sample preservative solution must be taken.

6.5.3.4. If samples are known or expected to contain target analytes over a wide range of concentrations, thereby requiring the analyses of multiple sample aliquots, it may be advisable and practical to take an additional sample aliquot in a low concentration soil vial, but collecting only 1-2 g instead of the 5 g typically collected. This aliquot may be used for those analytes that exceed the instrument calibration range in the 5g analysis.

6.5.3.5. Package, ship, and store samples as required in the table above.

6.6. High concentration soil samples - Soil samples to be analyzed using a methanol extraction or methanol-preserved sample. The methanol preservation portion of this procedure includes filling and weighing of vials in the field. As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

6.6.1. Collect the sample according to the procedures outlined in the sampling plan. Care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Always wear gloves whenever handling the tared sample vials. Preferred high concentration sample collection and preservation options are as follows:

Collection	Preservation	Holding Time	Comment
Encore®, or similar, coring device	Sample is extruded into a vial containing methanol and cooled to 4 ± 2°C.	14 days	
Bulk soil sampling jar.	No headspace, cool to 4 ± 2°C.	14 days	For projects using 5030A type sampling.

6.6.2. Preparation of VOA vials used in the collection of high concentration samples

6.6.2.1. Add methanol to allow a 1:1 ratio of soil/methanol (typically 5 mL methanol for 5g soil). Add the methanol to each vial.

6.6.2.2. Seal the vial with the screw-cap and septum seal.

6.6.2.3. Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label.

6.6.2.4. Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

6.6.3. If high concentration samples are collected without methanol preservation, a variety of sample containers may be employed, including 2 or 4 ounce glass vials with septum seals.

6.6.4. Sample collection

6.6.4.1. Using an appropriate sample coring or collection device, collect approximately 5g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most.

6.6.4.2. Carefully wipe the exterior of the sample collection device with a clean cloth or towel. Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the vial containing methanol. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap.

6.6.4.3. If high concentration samples are collected in vials containing methanol, then two aliquots should be collected, one for high concentration analysis collected in a vial containing methanol, and another for the dry weight determination in a vial without methanol.

6.6.4.4. Package, ship, and store samples as required in the table above. Shipments must be clearly labeled as containing methanol.

6.6.4.5. The collection of high concentration soil samples that are not preserved in the field generally follows the methanol-preserved procedure, with the exception that the sample vials contain no preservative. Fill the sample container as full as practical in order to minimize the headspace. It is preferable to collect a second sample aliquot.

6.7. Waste samples – For waste samples soluble in methanol, samples should be collected using one of the following options:

Collection	Preservation	Holding Time	Comment
VOA vial, with minimal headspace	Minimal headspace, cool to 4 ± 2°C.	14 days	
Bulk soil sampling jar.	Minimal headspace, cool to 4 ± 2°C.	14 days	For projects using 5030A type sampling.

6.7.1. Sample containers for waste samples require no additional preparation.

6.7.2. Sample collection

6.7.2.1. Using an appropriate sample collection device, fill the vial with the waste sample such that minimal headspace if left.

6.7.2.2. Carefully wipe the exterior of the vial and threads with a clean cloth or towel and immediately seal the vial with the septum and screw-cap.

6.7.2.3. Package, ship, and store samples as required in the table above.

6.8. Multi-Increment Soil Sampling - GRO, BTEX, Volatile Organic Contaminants

6.8.1. Samples for volatile analyses must be collected before non-volatiles to reduce contaminant losses due to volatilization. Collect the much smaller increment for

volatile analyses directly into the sample jar that contains the methanol. This is followed by the collection of the larger scoop to be sieved for non-volatile analyses.

6.8.2. Labile organics require that samples be preserved with a minimum 1:1 ratio of sample preservative to sample material (1 ml methanol to 1 gram (g) soil). This is a minimum required ratio, and additional sample mass is preferred as long as it is completely submerged by the methanol. The concern with MI is that the collection and sieving of the sample material will lead to volatilization of the contaminants, so sieving must not be performed for any volatile analyses (GRO, BTEX, or VOCs).

6.8.3. Samples for volatile analysis should be received prepared from the field in the appropriate containers. In cases where proper field sampling does not occur the following steps will be taken.

6.8.4. Begin by placing the appropriate amount of methanol into the sample container. When sampling from a bucket, be sure to sample from the center and remove a few inches of soil. Use caution to ensure that the sample increment selected represents soil from the desired depth and not "sluff" material from an upper level. To the extent possible, the individual increments should consist of the smaller particles (< 2millimeter (mm)) to be similar to the non-volatile sieved sample matrix and to minimize FE. Large rocks or clumps of soil must not be collected as part of the sampling of volatiles, as this will increase the sampling error.

6.8.5. The volatile sampling procedure should be accomplished as quickly as possible to reduce the loss of soil contaminants and methanol due to volatilization. Care must also be taken to prevent the loss of methanol due to splashing during the addition of soil increments and/or spillage during the entire sampling procedure.

6.9. Sample shipment and handling

6.9.1. Unless a freezing sample preservation option is being used, all samples for volatiles analysis should be cooled to approximately 4 ± 2°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan.

6.9.2. Once in the laboratory, samples are stored at the designated temperature until analysis or preservation. The sample storage area should be free of organic solvent vapors.

6.9.3. All samples should be analyzed as soon as practical, and within the designated holding time from collection.

6.9.4. When the low concentration samples are received with only Encore®, or similar, samples included, one core sample per lab code is immediately methanol preserved. That sample is transferred from the Encore® to a VOA vial containing a 5mL aliquot of methanol and the sample weight recorded. The vial is vortexed for 30 seconds and left to settle for a minimum of 15 minutes. The methanol is then decanted into a small

vial to minimize headspace. A cap with septum is then secured to the vial. The extract must be stored in the dark at 4 ± 2°C until analysis. A Preparation Blank using the same methanol lot number is created at the time of the preservation. The Encore® samples not used for methanol preservation must either be preserved for low-level analysis (using one of the method options) or analyzed or within 48 hours of collection. Refer to Table 1 for a complete list of holding times.

7. APPARATUS AND MATERIALS

7.1. Purge-and-Trap System - The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards (if applicable) to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph.

7.1.1. Sample Containers – The Archon autosampler uses 40mL clear vials equipped with the PTFE-faced silicone septa.

7.1.2. The Archon is capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 5 mL of organic-free reagent water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means. The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph.

7.1.3. The purge-and-trap device consists of three separate pieces of equipment: the sample purger, the trap, and the desorber. A Tekmar LSC 2000, 3000, 3100 or equivalent is used. Refer to Figures 1-4 in Method 5030B for typical configurations.

7.1.4. The recommended purging chamber is designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria. Alternate sample purge devices may be used, provided equivalent or improved performance is demonstrated.

7.1.5. The traps used for the determinative methods are listed below (alternate purge-and-trap devices may use traps of different types or manufacture as long as equivalent or improved performance is demonstrated).

- Method 8260,8021(PID/Hall): Supelco "K" trap
  part number 2-4920-V(Tekmar) or 2-1131-V(O.I.)

- Method 8015,8021(FID/PID): Supelco "J" trap
  part number 2-4919 (Tekmar) or 2-1145 (O.I.)

7.1.6. The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas.

7.1.7. Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

7.2. Syringes and Syringe Valves

7.2.1. 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

7.2.2. 25-µL micro syringe with a 2 inch x 0.006 inch ID, 22E bevel needle (Hamilton #702N or equivalent).

7.2.3. Micro syringes - 10-, 100-µL.

7.2.4. Syringes - 0.5-, 1.0-, and 5-mL, gas-tight.

7.3. Glass vials - 60-mL, septum-sealed, to collect samples for screening, dry weight determination.

7.4. Vials - 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

7.5. Top-loading balance - Capable of accurately weighing to 0.01 g.

7.6. Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

7.7. Volumetric flasks - Class A, 10-mL and 100-mL, with ground-glass stoppers.

7.8. Volumetric flasks - Class A, 10-mL and 100-mL, with ground-glass stoppers.

7.9. Vials - 2 mL glass, for GC autosampler - Used for oily waste samples extracted with methanol.

7.10. Spatula, stainless steel - narrow enough to fit into a sample vial.

7.11. Disposable Pasteur pipettes.

7.12. Field Sampling Equipment

7.12.1. Purge-and-Trap Soil Sampler - Model 3780PT (Associated Design and Manufacturing Company, Alexandria, VA 22314), or equivalent.

7.12.2. EnCore™ sampler - (En Chem, Inc., Green Bay, WI 54302) or equivalent.

7.12.3. Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

7.13. Portable balance - For field use, capable of weighing to 0.01 g.

7.14. Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, reagent water added, cap, and septum.

8. REAGENTS

8.1. Organic-free reagent water - All references to water in this method refer to organic-free reagent water.

8.2. Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents.

8.3. Low concentration sample preservative

8.3.1. Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

8.3.2. The preservative should be added to the vial prior to shipment to the field, and must be present in the vial prior to adding the sample.

8.4. Refer to the determinative method for details regarding preparation of standards and internal standard/surrogate spiking solutions.

9. RESPONSIBILITIES

9.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training

program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

9.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the determinative procedure is also the responsibility of the department supervisor/manager.

9.3. This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

10. PREVENTIVE MAINTENANCE

10.1. Carrier gas - Inline purifiers or scrubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

10.2. Due to high temperatures associated with the use of the traps described, care must be taken to asses trap breakdown and contamination on a daily basis. Both Supelco traps "J" and "K", under daily use, may not maintain performance for longer than 45 days. Traps used for 'short' analyte lists may last longer.

10.3. Samples lines in the autosampler and purge-and-trap unit along with the transfer line to the gas chromatograph have shown susceptibility to contamination and formation of active sites that may interfere with analysis. Daily assessment, through analysis of standards and blanks, needs to be conducted to determine need for maintenance before sample analysis begins.

10.4. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

11. PROCEDURE

NOTE: For soil/solid samples requiring VOA and non-VOA analyses and only one container was submitted to the lab, sample receiving will label the sample container as "VOA Analysis First" and/or attach a "VOA FIRST" tag. The VOA department will remove a sample aliquot first for their analyses. The sample should be handled as if it were a Rush analysis, so that the other non-VOA analyses will not be unduly delayed. The VOA analyst who opens the container will either break the custody seal and will initial and date it when the container was opened or sign and date the "VOA FIRST" tag. A VOA Analysis First note will also be included on the SR.

11.1. This section describes procedures for sample screening, the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

11.2. Sample screening

11.2.1. It is highly recommended that all samples be screened prior to the purge-and-trap GC or GC/MS analysis. Samples may contain higher than expected quantities of purgeable organics that will contaminate the purge-and-trap system, thereby requiring extensive cleanup and instrument maintenance. The screening data are used to determine which sample preparation procedure or dilution is the appropriate for the particular sample. For GC/MS analyses, all soil samples are screened.

11.2.2. When screening samples, refer to the SOP for *Sample Screening for Volatile Organic Compounds in Soil, Water, and Miscellaneous Matrices* (VOC-BVOC) for specific procedures.

11.2.3. Use the low concentration closed-system purge-and-trap procedure if the estimated concentration from the screening procedure falls within the calibration range of the selected determinative method. If the concentration exceeds the calibration range of the low concentration soil method, then use either the high concentration soil procedure or the oily waste procedure.

11.3. Instrument setup and calibration

11.3.1. Assemble the purge-and-trap device that is connected to a gas chromatograph or a gas chromatograph/mass spectrometer system.

11.3.2. For trap conditioning follow the manufacturer's recommendations. Do not vent the trap effluent to the analytical column. Prior to daily use, the trap should be conditioned for 10 minutes at 260°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

11.3.3. Prepare and analyze initial calibration standards as prescribed by the determinative method. The calibration standards are prepared in organic-free reagent water and analyzed the same as samples using one of the procedures listed for samples below. The volume of organic-free reagent water used for calibration must be the same volume used for sample analysis. The internal standard solution is added automatically, by the instrument, in the same fashion as used for the samples. Place the soil vial containing the solution in the instrument autosampler. In order to calibrate the surrogates using standards at five concentrations, it may be necessary to disable the automatic addition of surrogates to each vial containing a calibration standard (consult the manufacturer's instructions). Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as prescribed by the manufacturer.

11.4. Low concentration soil procedure. This procedure is used for the "direct-purge" analysis of soil samples collected and preserved accordingly (see Section 6).

11.4.1. The soil vial is hermetically sealed at the sampling site, and MUST remain so in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the exterior of the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

11.4.2. Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument autosampler according to the manufacturer's instructions. When transferring a sample aliquot from an Encore® sampler, a VOA vial containing a stir bar should be labeled and ready to receive the sample. The sample is then transferred from the Encore® (if frozen, transfer while still frozen to minimize loss of VOCs). Record the weight of the sample aliquot on a bench sheet and add 5 mL of reagent water. Seal with a cap and septum.

11.4.3. Without disturbing the hermetic seal on the sample vial, add 5 mL of organic-free reagent water, the internal standards, and the surrogate compounds. This is carried out using the autosampler. Other volumes of organic-free reagent water may be used, however, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free reagent water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes.

11.4.4. Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means.

11.4.5. After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/minute for about two minutes (1.5 min is normally adequate for analytes in Method 8015). Begin the temperature program of the gas chromatograph and start data acquisition.

11.4.6. After desorbing the sample for ~2 minutes, recondition the trap by stepping the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

11.4.7. If the concentration of any target analyte exceeds the calibration range of the instrument, it will be necessary to reanalyze the sample by the high concentration

method. Such reanalyses need only address those analytes for which the concentration exceeded the calibration range of the low concentration method. Alternatively, if a sample aliquot of 1-2 g was also collected it may be practical to analyze that aliquot for the analytes that exceeded the instrument calibration range in the 5g analysis.

11.5. High concentration soil procedure. The high concentration method is performed using a methanol extraction or methanol-preserved sample. An aliquot of the extract is added to organic-free reagent water (containing matrix spiking standards where applicable), purged, and analyzed by the selected determinative method. The autosampler is used to add internal standards and surrogates. The specific sample preparation steps depend on whether or not the sample was preserved in the field. Refer to Section 6 for preservation of samples that were not preserved in the field.

11.5.1. When the high concentration sample is not preserved in the field, the sample must first be extracted in methanol. If methanol preserved, skip to 11.5.2.

NOTE: This step must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

11.5.1.1. The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Whenever practical, mix the contents of the sample container by shaking or other mechanical means without opening the vial. When shaking is not practical, quickly mix the contents of the vial with a narrow metal spatula and immediately reseal the vial.

11.5.1.2. Prepare a VOA vial by adding 5mL of methanol to the labeled vial. Tare the vial. Quickly remove ~5g of the sample and add to the vial and record the weight of the sample. Quickly cap the vial and vortex for 30 seconds. Let stand for a minimum of 15 minutes.

11.5.1.3. Pipet the methanol from into a storage container that allows for no or minimal headspace and seal the container. Add methanol to a storage container of the same type for later use as a method blank. The extracts must be stored at 4 ± 2°C in the dark, prior to analysis.

11.5.2. For soil and solid waste samples that were preserved in methanol, weigh the vial to 0.01g as a check on the weight recorded in the field. Surrogate is added by the autosampler. Some sampling procedures require surrogate to be spiked in the field. Surrogate solution must not be spiked into samples already containing surrogate.

11.5.3. To analyze methanol-preserved samples or methanol extracts, add 500uL of the methanol to reagent water in a 50mL graduated cylinder and adjust to 50 mL (screening or analysis data may be used to determine other appropriate aliquots). In the case of methanol preserved samples, a low level analysis will typically exist to help

indicate the proper dilution level necessary. Analyze by Method 5030 in conjunction with the appropriate determinative method.

11.5.4. If the analysis shows a surrogate recovery outside of the acceptance criteria a reanalysis should be performed to confirm matrix interferences. Confirmation of matrix interferences or other possible sampling problems/limitations affecting surrogate recovery need to be documented in the case narrative. If the solution shows an unacceptable surrogate recovery a re-extraction should be performed if sufficient sample remains.

11.5.5. If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample, using the SOP for Total Solids., after the sample extract has been transferred to a GC vial and the vial sealed.

11.5.6. The water contained in the native sample is accounted for when determining the final extract volume. The final volume of the methanol extract is the total volume of the methanol/water mixture. Calculate the final volume as follows:

$$FinalVolume\ Methanol/Water = mL\ of\ solvent + \left(\frac{\%Moisture\ x\ Sample\ Wt.(g)}{100} \right)$$

11.6. High concentration soluble waste procedure. This procedure involves the dilution of the sample in methanol. Wastes that are insoluble in methanol and not analyzed by the methanol extraction procedure are diluted with hexadecane. Care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 50 mL of organic-free reagent water, purged, and analyzed using an appropriate determinative method.

11.6.1. Screening is highly recommended for product samples soluble in methanol. Samples soluble in methanol are often very high in matrix interferences or target compounds and should be diluted to minimize possible instrument damage. The most concentrated this extract can be analyzed is 1:100 (500 uL to 50 mL). A 1:5000 dilution is a better place to start if screening is not possible.

11.6.2. If the waste was not preserved in the field and it is soluble in methanol, weigh 1 g (wet weight) of the sample into a tared 10-mL volumetric flask.

11.6.3. Dilute to volume with methanol and invert the flask three times to mix the contents. Transfer the sample to a 7mL septa-sealed vial. Store in the dark at 4 ± 2°C. Surrogate and internal standard will be added by the autosampler.

11.6.4. If the sample was collected in the field in a vial containing methanol, weigh the vial to 0.01g as a check on the weight recorded in the field.

11.6.5. Regardless of how the sample was collected, the target analytes are soluble in the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

11.6.6. Add 500 µL of the methanol extract to 50 mL of organic-free reagent water for purge-and-trap analysis. For field-extracted samples, no methanol blank is stored with the samples.

12. QUALITY CONTROL

12.1. Before processing any samples, the analyst should demonstrate through the analysis of an organic-free reagent water method blank that all glassware and reagents are interference free.

12.2. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

12.3. Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. Refer to the determinative procedure for the information on how to accomplish this demonstration.

12.4. Sample Quality Control for Preparation and Analysis - See Sec. 8.0 in Method 5000 and Method 8000 for procedures to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

12.5. Refer to the SOP for the determinative method and the *SOP for Sample Batches* for minimum QC requirements.

13. DATA REDUCTION AND REPORTING

13.1. Record all weights and volumes used in the sample preparation steps described within this SOP on the applicable benchsheet or runlog. Note if samples were not collected following one of the sampling options so that the method reference is reported correctly.

13.2. Refer to the determinative procedure for the information on data reduction and reporting.

14. METHOD PERFORMANCE

This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

The method detection limit (MDL) is established using the procedure described in the SOP for The Determination of Method Detection Limits (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

15. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

16. WASTE MANAGEMENT

16.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

16.2. This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

17. TRAINING

17.1. Training Outline

17.1.1. Review literature by reading references. Review state specific methods for Gasoline Range Organics and Total Petroleum Hydrocarbons as Gasoline. Review the SOP. Also review MSDS for methanol.

17.1.2. Observe the procedure as performed by an experienced analyst at least three times.

17.1.3. Assist in the procedure under the guidance of an experienced analyst (for 1-3 months). During this training process, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.1.4. Following the training period the analyst is expected to complete applicable initial precision and recovery (IPR) studies. Summaries of the IPR are reviewed and signed by the supervisor and forwarded to the employee's training file.

17.1.4.1. Perform IPR studies by preparing and analyzing four replicate laboratory control samples spiked at a level of 10-20 times the MRL. Calculate average percent recovery and relative standard deviation for the four replicate analyses. Refer to Method 8000B for evaluation guidelines.

17.1.4.2. For applicable tests, IPR studies should be performed in order to be equivalent to NELAP Initial Demonstration of Capability.

17.2. Training is documented following the *SOP for Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. REFERENCES

18.1. EPA SW-846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 5035, Revision 0; and Method 5035A, July 2002.

18.2. SOP for Sample Screening for Volatile Organic Compounds in Soil, Water, and Miscellaneous Matrices (VOC-BVOC)

18.3. SOP for Bottle Order Preparation and Shipping (SMO-BORD).

18.4. Draft Guidance on Multi-Increment Soil Sampling State of Alaska, Department of Environmental Conservation, March 2007

18.5. Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples, EPA/600/R-03/027, November 2003

Summit Environmental Technology SOPs

S-1

SUMMIT ENVIRONMENTAL TECHNOLOGIES, INC

STANDARD OPERATING PROCEDURE FOR

METHOD 8082
POLYCHLORINATED BIPHENYLS (PCBs)
BY GAS CHROMATOGRAPHY

Approved by: _____
Laboratory Manager

Approved by: _____
Quality Manager

1.0 SCOPE AND APPLICATION

1.1 Method 8082 is used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors in extracts from solid and aqueous matrices. This method analyzes for the Aroclors listed below:

Compound	CAS Number
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 Compound identification based on single-column analysis is confirmed on a second column. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm the measurements made with the primary column.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1 L for liquids, 30 g or 2 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 3510C (separatory funnel).

2.3 Solid samples are extracted with Methylene chloride-acetone (1:1) using Method 3550B (Sonication).

2.4 The extract is analyzed by injecting a 2-μL aliquot into a gas chromatograph with a narrow or wide-bore fused silica capillary column and electron capture detector (GC/ECD).

3.0 DEFINITIONS

3.1 Refer to the Definition SOP for a listing of terms used in the laboratory.

4.0 INTERFERENCES

4.1 Sources of interference in this method can be grouped into three broad categories.

4.1.1 Contaminated solvents, reagents, carrier gas, GC parts, or sample processing hardware.

4.1.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

4.1.3 Compounds extracted from the sample matrix to which the detector will respond.

4.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations.

4.2.1 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.

4.2.2 Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2.3 These materials can be removed through the use of Method 3665A (sulfuric acid/permanganate cleanup).

4.3 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.

4.4 Elemental sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs.

5.0 SAFETY

5.1 All personnel performing this method wear lab coats, eye protection, and gloves.

5.2 All mixing and handling of reagents should be performed in a hood, if possible.

5.3 Each compound or reagent used in this method must be treated as a potential health hazard. Exposure to these compounds must be reduced to the lowest possible level. A current awareness file of OSHA regulations is maintained regarding the safe handling of the chemicals specified in this method. A reference file of materials safety data sheets (MSDSs) is made available to all personnel involved in this analysis.

5.4 Samples that may contain high concentrations of toxic compounds are handled with gloves and in a hood to prevent exposure.

6.0 EQUIPMENT AND SUPPLIES

6.1 Gas chromatograph -HP 5890/ECD with data system and autosampler

6.2 GC columns

6.2.1 Column 1: 30 m x 0.53 mm ID fused silica capillary column (DB-608), 0.83 µm film thickness. J & W Cat # 1257730 or equivalent.

6.2.2 Column 2: 30 m x 0.53 mm ID fused silica capillary column (RTx-5) 0.5 µm film thickness. Restek cat. # 10240 or equivalent.

6.3 Volumetric flasks - 10-mL and 25-mL, for preparation of standards if needed.

6.4 Spatula - Stainless steel or PTFE.

6.5 Syringe - 1 mL.

6.6 Consult SOPs for Methods 3510C and 3550B for necessary equipment for sample extraction.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade or pesticide grade chemicals must be used in all tests.

NOTE: Store the standard solutions (stock, composite, calibration, and surrogate standards) at 4°C in Polytetrafluoroethylene (PTFE)-sealed containers in the dark. 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.

7.2 Sample extracts prepared by Methods 3510C and 3550B, need to undergo a solvent exchange step prior to analysis. The following solvent is necessary for dilution of sample extracts. All solvent lots must be pesticide quality or equivalent and must be determined to be phthalate-free through method blank analysis.

7.2.1 n-Hexane, C_6H_{14}

7.3 Organic-free reagent water - All references to water in this method refer to organic-free reagent water.

7.4 The following solvents may be necessary for the preparation of standards.

5.3.1 Acetone, $(CH_3)_2CO$

5.3.2 Toluene, $C_6H_5CH_3$

7.5 Stock standard solutions (1000 mg/L) - Purchased as certified solutions.

Compound	Mfg.	Cat. No	Concentration µg/mL
Aroclor 1016	Restek	32006	1000
Aroclor 1221	Restek	32007	1000
Aroclor 1232	Restek	32008	1000
Aroclor 1242	Restek	32009	1000
Aroclor 1248	Restek	32010	1000
Aroclor 1254	Restek	32011	1000
Aroclor 1260	Restek	32012	1000

7.6 Calibration standards for Aroclors

7.6.1 Prepare an initial calibration standard of a minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260 as follows:

Level	1016 (µL)	1260 (µL)	Dilute to	Concentration (mg/L)
1	0.2	0.2	1 mL	0.2
2	1.0	1.0	1 mL	1.0
3	2.0	2.0	1 mL	2.0
4	4.0	4.0	1 mL	4.0
5	5.0	5.0	1 mL	5.0
6	10.0	10.0	1 mL	10.0

7.6.2 Prepare a 4 mg/L continuing calibration standard containing a mixture of Aroclor 1016 and Aroclor 1260 by adding 4 µL of 1000 mg/L standard that is obtained from a second source or from a different lot number than the initial calibration mixture into 1 mL of hexane.

7.6.3 Single standards of each of the other five Aroclors are used to aid in pattern recognition, these single standards of the remaining five Aroclors are also used to determine the calibration factor for each Aroclor. Prepare a standard for each of the other Aroclors as follows:

Aroclor	µL	Dilute to	Concentration (mg/L)
1221	5.0	1 mL	5.0
1232	5.0	1 mL	5.0
1242	5.0	1 mL	5.0
1248	5.0	1 mL	5.0
1254	5.0	1 mL	5.0

7.7 Surrogate standards

7.7.1 2,4,5,6-Tetrachloro-m-xylene (TCMX) is used as a surrogate. Prepare a stock solution of TCMX at a concentration of 100 mg/L in hexane by weighing 0.01gm of TCMX into 100 mL flask and dilute to volume with hexane.

7.7.2 Surrogate Spiking Solution: ~~use~~ dilute 1:10 the stock solution above to make surrogate spiking solution at a concentration of 1 mg/L. One mL of this solution is added to all samples, blanks, MS/MSD and LCS/LCSD prior to extraction. The surrogate solution is also added to all calibration standards.

7.8 Matrix Spiking Solution

Matrix spiking solutions are purchased as certified standards. Aroclor 1016 and 1260 are used for MS/MSD and LCS or LCSD spikes.

Aroclor	Mfg.	Cat. #	Conc. mg/L	µL used
1016	Supelco	48097	1000	10
1260	Supelco	44808	1000	10

7.9 DDT/Endrin standard - Used to determine if DDT elutes at the same retention times as any of the target analytes (Aroclors).

7.9.1 The DDT/Endrin stock standard mix is obtained from Supelco (Stock No. 4-8282) at a concentration of 500 µg/mL. Add 1 µL of the mix to 1 mL of hexane to obtain a 0.5 µg/mL concentration.

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

8.1.1 Water samples must be extracted within 7 days from time of collection and analyzed within 40 days from time of extraction. Soil/solids samples must be extracted within 14 days from time of collection and analyzed within 40 days from extraction time.

9.0 QUALITY CONTROL

9.1 Initial demonstration of proficiency

9.1.1 Each analyst must demonstrate initial proficiency with each sample preparation and determinative method combination he/she utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The following operations are performed whenever new analyst is trained or significant changes in instrumentation are made.

9.1.2 A QC Reference Sample concentrate (LCS) must contain PCBs as Aroclors at 10-50 mg/L in the concentrate for water samples. A 1-mL volume of this concentrate spiked into 1 L of reagent water will result in a sample concentration of 10-50 µg/L. If Aroclors are not expected in samples from a particular source, then prepare the QC reference samples with a mixture of Aroclors 1016 and 1260. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclors must be used for the QC reference sample.

9.1.3 Calculate the average recovery and the standard deviation of the recoveries of the analytes in each of the four QC reference samples.

9.2 Sample Quality Control for Preparation and Analysis - The analysis of QC samples including a method blank, a matrix spike, a matrix spike duplicate (MS/MSD), and a laboratory control sample (LCS) in each analytical batch (up to 20 samples) and the addition of surrogates to each field sample and QC samples is required.

9.2.1 A method blank must be extracted with every extraction batch, up to 20 samples.

9.2.1.1 Prepare a method blank by extracting the same amount or volume of clean matrix as samples.

9.2.2 Matrix Spike and Matrix Spike Duplicate samples must be extracted with every batch.

9.2.2.1 Prepare a matrix spike sample by spiking a second aliquot of a field sample as described in section 7.8. Prepare a matrix spike duplicate sample from a third aliquot of the sample.

9.2.2.2 If samples are not expected to contain target analytes, 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 must be used for spiking. If samples are expected to contain target analytes, then use one matrix spike and a duplicate analysis of an unspiked field sample.

9.2.3 A Laboratory Control Sample (LCS) must be included with each analytical batch. 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.

NOTE: An LCS duplicate (LCSD) may be used if insufficient sample is provided for and MS/MSD.

9.3 Surrogate recoveries - Evaluate surrogate recovery data from individual samples versus surrogate control limits.

9.4 Include a calibration standard after each group of 10 samples 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, stop analyses and take corrective action.

10.0 CALIBRATION AND STANDARDIZATION

10.1 External Standard Calibration Technique:

10.1.1 Prepare calibration standards (including surrogates) at a minimum of five different concentrations as described in sec. 7.6.

10.1.1.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. 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 multi-point calibration is performed using the mixture of Aroclors 1016 and 1260 prepared.

10.1.1.2 Standards of the other five Aroclors are necessary for pattern recognition. When employing the traditional model of a linear calibration through the origin, 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 standards for these five Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the five 1016/1260 standards.

10.1.1.3 In situations where only a few Aroclors are of interest for a specific project, 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.

10.1.2 Assemble the necessary GC apparatus and establish operating parameters equivalent to those indicated in Section 11.3.

NOTE: Once established, the same operating conditions must be used for both calibrations and sample analyses.

10.1.3 A 2 µL injection of each calibration standard is used.

10.1.4 Record the peak area for each characteristic Aroclor peak to be used for quantitation.

10.1.4.1 At least ten of the peaks are chosen for each Aroclor. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set peaks should include at least one peak that is unique to that Aroclor. Use at least 11 peaks for the Aroclors 1016/1260 mixture, none of which should be found in both of these Aroclors.

10.1.5 Use external standard technique to calculate the calibration factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards using the equation below.

$$CF = \frac{\text{Peak Area (or Height) in the Standard}}{\text{Total Mass of the Standard Injected (in nanograms)}}$$

10.1.6 The calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. This involves the calculation of the mean response or calibration factor, the standard deviation, and the relative standard deviation (RSD) for each Aroclor peak.

10.1.7 To evaluate the linearity of the initial calibration, calculate the mean CF ($\overline{CF}$), the standard deviation (SD), and the relative standard deviation (RSD) as follows:

$$\text{Mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{n-1}}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

Where N is the number of calibration standards. If the RSD of the calibration factor is less that or equal to 20% over the calibration range, the linearity through the origin may be assumed, and the average calibration factor may be used to determine sample concentration.

or

10.1.8 A linear regression calibration can be used for an external calibration.

This is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention.

The regression will produce the slope and intercept terms for a linear equation in the form:

$$Y = ax + b$$

Where:
y = Instrument response (peak area or height)
a = Slope of the line (also called the coefficient of x)
x = Concentration of the calibration standard
b = The intercept

Do not force the line through the origin, but have the intercept calculated from the five data points. Otherwise, the problems noted with the RSD value will occur, i.e., a line through the origin will not meet the QC specifications. In addition, do not include the origin (0,0) as a sixth calibration point. The use of a linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995.

In calculating sample concentrations by the external standard method, the regression equation is rearranged to solve for the concentration (x), as shown below.

$$X = \frac{(y - b)}{a}$$

10.3 Calibration Verification

10.3.1 The working calibration curve and retention times must be verified at the beginning of each 12-hour work shift as a minimum requirement. Verification is accomplished by the measurement of verification standard. Additional analyses of the verification standard(s) throughout a 12-hour shift are required. Calibration verification standard must contain surrogate.

10.3.1 Continuing calibration verifications (CCV) must meet the ± 10% acceptance limits. Samples analyzed prior to a failed CCV must be re-analyzed.

10.3.2 Check instrument conditions and procedures to determine the cause of the problem. Correct any problems and re-analyze the CCV. If CCV passes, re-analyze the samples analyzed previous to the failed CCV.

10.33 If CCV again fails acceptance limits, recalibrate the instrument and re-analyze samples.

10.3.2 Calculate the % difference as follows:

$$\% \text{ Difference} = \frac{CF_v - \overline{CF}}{\overline{CF}} \times 100$$

Where:

CF_v is the calibration factor from the analysis of the verification standard

$\overline{CF}$ is the mean calibration factor from the initial calibration.

The % difference calculated for the calibration verification standard must be within ±15% for each analyte, before any sample analyses may take place.

10.3.3 If the retention time of any analyte does not fall within the ± 3σ window, corrective action must be taken to restore the system or a new calibration curve must be prepared.

10.3.4 Solvent blanks and any method blanks must be run with calibration verification analyses (every 12 hours) to confirm that laboratory contamination does not cause false positives.

10.3.5 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with calibration verification followed by sample extract analyses. Additional analyses of the verification standard(s) throughout a 12-hour shift are required. A verification standard is also necessary at the end of a set. The sequence ends when the set of samples has been injected or when retention time and/or % difference QC criteria are exceeded.

If the criteria are exceeded, inspect the GC system to determine the cause and perform whatever maintenance is necessary before recalibrating and proceeding with sample analysis. All samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable.

10.3.6 The performance of the entire analytical system must be checked every 12 hours, using data gathered from analyses of blanks, standards, and replicate samples. Significant peak tailing must be corrected.

10.3.7 Prepare calibration standards as described in sec. 7.6.

10.3.7.1 In situations where only a few Aroclors are of interest for a specific project, 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.

10.4 Retention time windows

10.4.1 Retention time windows (RTW) must be established prior to sample analysis. Refer to the Method 8000 SOP for instructions concerning RTW determinations.

10.4.2 Retention time windows are verified prior to each sample analysis sequence or each 12 hour shift. Retention time windows must be re-established prior to further sample analysis if acceptance criteria is not met.

16.4.3 The retention time of all analytes in all verification standards must fall within the absolute RT windows. If an analyte falls outside the RT window in a calibration verification standard, new absolute RT windows must be calculated, unless instrument maintenance corrects the problem.

10.4.4 Before establishing retention time windows, make sure that the chromatographic system is operating reliably. Make three injections of all multi-component analytes over the course of a 72-hour period.

10.4.5 Record the retention time for each specific Aroclor peak, and calculate the mean and standard deviation of those peaks.

10.4.6 The width of the retention time window for each major peak is defined as +/- 3 times the standard deviation of the mean absolute retention time established during the 72-hour period.

10.4.7 New retention time windows must be established when a new GC column is installed.

10.4.8 See Table 1 for example retention time windows of Aroclors.

10.5 Any data found to be out of control or unacceptable that could not be corrected must be flagged with a proper explanation and indicated on the analytical report.

10.5.1 Notify the supervisor of the problem.

10.5.2 Contact the client to report any out of control or unacceptable data for resolution of the problem if possible. Re-sampling or client acceptance of the data may be options.

10.5.3 Initiate any relevant corrective action to alleviate the problem if possible.

11.0 PROCEDURE

11.1 Sample extraction

11.1.1 Water samples are extracted at a neutral pH with Methylene chloride using a separatory funnel (Method 3510C). Solid samples are extracted with methylene chloride-acetone (1:1) using, Sonication (Method 3550B). Refer to the individual SOPs for Methods 3510C and 3550B for details on extraction procedures.

11.3 Typical GC conditions

Carrier gas (Helium) flow rate:	5 mL/minute
Makeup gas (Nitrogen) flow rate:	30 mL/min
Injector temperature:	200°C
Detector temperature:	275°C
Temperature program:	
Initial temperature:	150°C, hold 2 minute
Program:	150°C to 230°C at 4°C/min
Final temperature:	230°C to 275°C, at 10°C/min
Final hold:	3 min

11.4 Gas chromatographic analysis of sample extracts

11.4.1 The same GC operating conditions used for the initial calibration must be employed for sample analyses.

11.4.2 Inject a 2-µL aliquot of the concentrated sample extract.

11.4.3 Qualitative identifications of target analytes are made by examination of the sample chromatograms.

11.5 Qualitative identification

11.5.1 The identification of PCBs as Aroclors using this method 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 that has been properly calibrated and retention time windows established, or GC/MS analysis if the PCB concentration is sufficient for analysis.

11.6 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.

11.6.1 Use the individual Aroclor standards to determine the pattern of peaks.

11.6.2 Once the Aroclor pattern has been identified compare the responses of at least 10 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 individual calibration factor for each of the characteristic peaks and the external calibration technique. A concentration is determined by adding individual concentrations and the average is used to determine concentration of that Aroclor.

11.6.3 Using external calibration procedure, determine the quantity of each Aroclor in the sample

11.6.3.1 For Aqueous samples, determine the concentration of an analyte as follows:

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(V_t)(D)}{(\overline{CF})(V_i)(V_s)}$$

Where:

A_s = Area of the peak for the analyte in the sample
V_t = Total volume of the concentrated extract (µL)
D = Dilution factor
CF = Mean calibration factor from the initial calibration
V_i = Volume of the extract injected (µL).
V_s = Volume of aqueous sample extracted in mL. If the units of liters are used for this term, multiply the results by 1000.

11.6.3.2 For Nonaqueous samples, determine the concentration of Aroclor as follows:

$$\text{Concentration } (\mu g/Kg) = \frac{(A_s)(V_t)(D)}{(CF)(V_i)(W_s)}$$

Where As, V_t, D, CF, and Vi are as described above.
W_s = Weight of sample extracted. If the units of kilograms are used for this term, multiply the results by 1000.

11.6.4 If the peak area exceeds the linear range of the system, dilute the extract and reanalyze.

11.6.5 Confirm the results on a second column that meets all calibration and retention time requirements.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Calculation of recovery

Calculate the recovery of each spiked analyte in the matrix spike, matrix spike duplicate and LCS according to the following formula.

$$\text{Recovery= \% R=} \frac{C_s - C_u}{C_n} \text{ X 100}$$

Where:

C_s = Measured concentration of the spiked sample aliquot
C_u = Measured concentration of the unspiked sample aliquot
C_n = Spiked Value

12.2 Calculation of precision

Calculate the relative percent difference (RPD) of the concentration measured for matrix spike/matrix spike duplicate pairs according to the formula below.

$$RPD = \frac{|C_1 - C_2|}{\frac{(C_1 + C_2)}{2}} \text{ X 100}$$

Where:

C_1 = Measured concentration of the first sample aliquot
C_2 = Measured concentration of the second sample aliquot

12.3 Calibration calculations are included in Section 10.

13.0 METHOD PERFORMANCE

13.1 Method detection limit (MDL) studies are conducted annually or when there are significant instrument changes. Current MDL studies are on file in the Quality Managers office. Refer to Table 3 for an example MDL study.

13.2 Reporting Limits - 0.2 mg/L for waters and 0.2 mg/kg for soils. Reporting limits may be changed according to client request.

13.3 Accuracy and Precision Data Refer to Table 2 for accuracy and precision data.

13.4 Consult Method 8082 for further information on method performance.

14.0 POLLUTION PREVENTION

14.1 The solvents and/or reagents used in this method pose little threat to the environment since they are either recycled or disposed of properly.

14.2 Standards are prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

15.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

15.1 Refer to Table 3 for information concerning data assessment and acceptance criteria.

16.0 CORRECTIVE ACTIONS AND CONTINGENCIES FOR UNACCEPTABLE DATA

16.1 Method Blank (BLK):

16.1.1 The results of the method blank must be less than 2 times the MDL.

16.1.2 If the method blank results do not meet the acceptance criteria above, then the analyst must take corrective action to locate and reduce the source of the contamination and re-extract or re-analyze all samples associated with the contaminated method blank.

16.1.3 Results of the method blank are not subtracted from samples results.

16.1.4 If there is evidence that carryover from a high-concentration sample into subsequent samples may have occurred, then samples are reanalyzed.

16.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD):

If percent recovery in the matrix spike or matrix spike duplicate falls outside the designated range for recovery, the analyst must determine if there is a matrix effect or a laboratory performance problem. A matrix effect is indicated if the LCS data are within limits but the matrix spike data exceed the limits.

16.3 Laboratory Control Sample (LCS) Laboratory Control Sample Duplicate (LCSD):

16.3.1 If percent recovery in the LCS falls outside the designated range for recovery perform the following:

16.3.1.1 Check to be sure that there are no errors in the calculations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas.

16.3.1.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze samples.

16.3.1.3 If, upon re-analysis, the recovery is again not within limits, report the data as an "Estimated Concentration". If the recovery is within the limits in the re-analysis, use the re-analysis data.

16.3.2 If an LCSD is analyzed:

16.3.2.1 If percent recovery in the LCS and LCSD falls outside the designated range for recovery and precision, then all samples associated with that LCS and LCSD must be re-extracted and re-analyzed.

16.4 Surrogates

16.4.1 Evaluate surrogate recovery data from individual field samples versus surrogate recovery limits. If recovery is not within laboratory surrogate recovery limits, then the following procedures are necessary:

16.4.1.1 Check to be sure that there are no errors in the calculations or surrogate solutions. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas.

16.4.1.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the sample.

16.4.1.3 If no instrument problem is found, the sample must be re-extracted and re-analyzed.

16.4.1.4 If, upon re-analysis, the recovery is again not within limits, report the data as an "Estimated Concentration". If the recovery is within the limits in the re-analysis, use the re-analysis data.

16.4.2 Evaluate surrogate recovery for QC samples. If recovery is not within laboratory surrogate recovery limits. The following procedures are necessary:

16.4.2.1 If surrogate fails for the LCS, refer to the MS/MSD analysis, follow the instructions in sections 16.3.1.1 and 16.3.1.2. If the MS/MSD surrogate and spike recoveries pass acceptance limits and all associated samples pass surrogate acceptance limits, the sample batch is considered acceptable.

16.4.2.2 If the surrogate fails for the MS/MSD analysis, refer to the LCS surrogate recovery. If the LCS surrogate recovery passes acceptance limits, the sample batch is acceptable. The MS/MSD is not re-extracted and re-analyzed.

16.4.2.3 If the surrogate recoveries for the LCS and the MS/MSD fail acceptance limits the associated analytical batch must be re-extracted and re-analyzed.

16.4.2.4 If the surrogate recoveries for the Method Blank fail acceptance limits the associated analytical batch must be re-extracted and re-analyzed

16.5 Initial Calibrations

16.5.1 Initial calibrations must meet method acceptance limits. Calibrations that fail acceptance limits must be repeated until acceptance limits are met.

16.5.2 Check instrument conditions or perform maintenance to eliminate any conditions that prevent a compliant initial calibration.

16.6 Continuing Calibrations

16.6.1 Continuing calibration verifications (CCV) must meet the ± 10% acceptance limits. Samples analyzed prior to a failed CCV must be re-analyzed.

16.6.2 Check instrument conditions and procedures to determine the cause of the problem. Correct any problems and re-analyze the CCV. If CCV passes, re-analyze the samples analyzed previous to the failed CCV.

16.6.3 If CCV again fails acceptance limits, recalibrate the instrument and re-analyze samples.

17.0 WASTE MANAGEMENT

17.1 The laboratory complies with all Federal, State, and local regulations governing waste management and land disposal restrictions. Protection of the air, water, and land are minimized and by controlled release from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required. Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner.

17.2 Samples preserved with acids to pH < 2 or caustics to a pH >12 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

17.3 Consult the laboratory Safety and Hygiene manual for further instruction.

18.0 REFERENCES

18.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Updates III, IVA, IVB, USEPA, 1200 Pennsylvania Ave. N.W., Washington, D.C., 20460.

19.0 TABLES AND DIAGRAMS

TABLE 1
SUMMARY OF RETENTION TIMES OF AROCLORS
ON THE RTX-5 COLUMN

	Aroclor 1016		Aroclor 1232		Aroclor 1242	
Peak #	RT mean	RT window	RT Mean	RT window	RT Mean	RT window
1	3.353	3.339-3.367	3.363	2.974-3.752	3.753	3.739-3.767
2	4.303	4.289-4.317	4.043	4.029-4.057	4.250	4.250-4.250
3	4.800	4.776-4.824	4.513	4.499-4.527	4.730	4.730-4.730
4	5.410	5.410-5.410	5.373	5.322-5.424	5.693	5.642-5.744
5	5.673	5.659-5.687	5.543	5.506-5.581	5.860	5.626-6.094
6	5.923	5.909-5.937	5.900	5.876-5.924	6.140	6.140-6.140
7	6.193	6.179-6.207	6.467	6.438-6.495	6.710	6.710-6.710
8	6.763	6.749-6.777	6.720	6.696-6.744	6.977	6.963-6.991
9	7.007	6.978-7.035	7.983	7.955-8.012	8.243	8.229-8.257
10	7.757	7.728-7.785	8.417	8.379-8.454	8.690	8.666-8.714
11	8.287	8.258-8.315				
	Aroclor 1248		Aroclor 1254		Aroclor 1260	
Peak #	RT mean	RT window	RT Mean	RT window	RT Mean	RT window
1	4.240	4.155-4.325	6.147	6.118-6.175	9.500	9.476-9.524
2	4.723	4.645-4.802	7.593	7.579-7.607	9.870	9.846-9.894
3	5.877	5.826-5.928	8.250	8.226-8.274	10.520	10.496-10.544
4	6.137	6.186-6.188	9.123	9.109-9.137	10.753	10.716-10.791
5	6.703	6.647—6.760	6.443	9.429-9.457	10.960	10.936-10.984
6	6.983	6.927-7.040	9.643	9.629-9.657	11.427	11.398-11.455
7	8.237	8.208-8.265	10.410	10.386-10.434	11.593	11.556-11.631
8	8.823	8.809-8.837	10.550	10.550-10.550	11.687	11.658-11.715
9	9.113	9.099-9.127	11.340	11.316-11.364	12.370	12.346-12.394
10	9.427	9.398-9.455	12.253	12.239-12.267	13.180	13.156-13.204
11			13.063	13.026-13.101	14.123	14.086-14.161
12					15.063	15.026-15.101
13					15.753	15.716-15.791

TABLE 2
QC LIMITS

Requirement	8082 (Water) Aroclor 1016	8082 (Soil) Aroclor 1016
Matrix Spike/Matrix Spike Duplicate	60-130	5-124
% RPD	0-30	0-30
Laboratory Control Sample/ Laboratory Control Sample Duplicate	60-130	50-124
Surrogate	19-93	17-104
Method Blank	<2 MDL	<2 MDL

Requirement	8082 (Water) Aroclor 1260	8082 (Soil) Aroclor 1260
Matrix Spike/Matrix Spike Duplicate	41-62	5-124
% RPD	0-30	0-30
Laboratory Control Sample/ Laboratory Control Sample Duplicate	60-130	50-124
Surrogate	19-93	17-104
Method Blank	<2 MDL	<2 MDL

TABLE 3
MDL STUDIES

Aroclor	Water MDL (µg/L)	Soil MDL (mg/Kg)
1016	0.20	0.006
1232	0.30	0.007
1242	0.24	0.009
1248	0.33	0.009
1254	0.25	0.009
1260	0.24	0.011

TABLE 4
REPORTING LIMITS

Aroclor	Water (µg/L)	Soil (mg/Kg)
1016	1.0	2.0
1232	1.0	2.0
1242	1.0	2.0
1248	1.0	2.0
1254	1.0	2.0
1260	1.0	2.0

S-2

Designation: D 445 – 01

An American National Standard
British Standard 2000: Part 71:1990

Designation: 71/1/97

Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)[1]

This standard is issued under the fixed designation D 445; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This test method has been approved by the sponsoring committee and accepted by the Cooperating Societies in accordance with established procedures.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method specifies a procedure for the determination of the kinematic viscosity, ν, of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity, η, can be obtained by multiplying the kinematic viscosity, ν, by the density, ρ, of the liquid.

NOTE 1—For the measurement of the kinematic viscosity and viscosity of bitumens, see also Test Methods D 2170 and D 2171.

1.2 The result obtained from this test method is dependent upon the behavior of the sample and is intended for application to liquids for which primarily the shear stress and shear rates are proportional (Newtonian flow behavior). If, however, the viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of different capillary diameters. The procedure and precision values for residual fuel oils, which under some conditions exhibit non-Newtonian behavior, have been included.

1.3 The range of kinematic viscosities covered by this test method is from 0.2 to 300 000 mm^2/s (see Table A1.1) at all temperatures (see 6.3 and 6.4). The precision has only been determined for those materials, kinematic viscosity ranges and temperatures as shown in the footnotes to the precision section.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers[2]

D 1193 Specification for Reagent Water[3]

D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer[2]

D 1480 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer[2]

D 1481 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer[2]

D 2170 Test Method for Kinematic Viscosity of Asphalts (Bitumens)[4]

D 2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer[4]

D 6074 Guide for Characterizing Hydrocarbon Lubricant Base Oils[5]

E 1 Specification for ASTM Thermometers[6]

E 77 Test Method for the Inspection and Verification of Thermometers[6]

2.2 *ISO Standards:*[7]

ISO Guide 25—General Requirements for the Calibration and Testing Laboratories

ISO 3104 Petroleum Products—Transparent and Opaque

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

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In the IP, this test method is under the jurisdiction of the Standardization Committee.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 04.03.

[5] *Annual Book of ASTM Standards*, Vol 05.03.

[6] *Annual Book of ASTM Standards*, Vol 14.03.

[7] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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Liquids—Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity
ISO 3105 Glass Capillary Kinematic Viscometers—Specification and Operating Instructions
ISO 3696 Water for Analytical Laboratory Use—Specification and Test Methods
ISO 9000 Quality Management and Quality Assurance Standards—Guidelines for Selection and Use

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *automated viscometer, n*—apparatus which, in part or in whole, has mechanized one or more of the procedural steps indicated in 10 or 11 without changing the principle or technique of the basic manual apparatus. The essential elements of the apparatus in respect to dimensions, design and operational characteristics are not changed. The measured result from the apparatus does not require correction to bring it into correlation with the basic manual apparatus. The precision of the apparatus shall be of statistical equivalence to, or better (has less variability) than the manual apparatus.

3.1.1.1 *Discussion*—Automated viscometers have the capability to mimic some operation of the test method while reducing or removing the need for manual intervention or interpretation. Apparatus which determine kinematic viscosity by physical techniques that are different than those used in this test method are not considered to be Automated Viscometers.

3.1.2 *density, n*—the mass per unit volume of a substance at a given temperature.

3.1.3 *dynamic viscosity, n*—the ratio between the applied shear stress and rate of shear of a liquid.

3.1.3.1 *Discussion*—It is sometimes called the coefficient of dynamic viscosity or, simply, viscosity. Thus dynamic viscosity is a measure of the resistance to flow or deformation of a liquid.

3.1.3.2 *Discussion*—The term dynamic viscosity can also be used in a different context to denote a frequency-dependent quantity in which shear stress and shear rate have a sinusodial time dependence.

3.1.4 *kinematic viscosity, n*—the resistance to flow of a fluid under gravity.

3.1.4.1 *Discussion*—For gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density, ρ. For any particular viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity, ν, where $\nu = \eta/\rho$, and η is the dynamic viscosity coefficient.

4. Summary of Test Method

4.1 The time is measured for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled and known temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

5. Significance and Use

5.1 Many petroleum products, and some non-petroleum materials, are used as lubricants, and the correct operation of the equipment depends upon the appropriate viscosity of the liquid being used. In addition, the viscosity of many petroleum fuels is important for the estimation of optimum storage, handling, and operational conditions. Thus, the accurate determination of viscosity is essential to many product specifications.

6. Apparatus

6.1 *Viscometers*—Use only calibrated viscometers of the glass capillary type, capable of being used to determine kinematic viscosity within the limits of the precision given in the precision section.

6.1.1 Viscometers listed in Table A1.1, whose specifications meet those given in Specifications D 446 and in ISO 3105 meet these requirements. It is not intended to restrict this test method to the use of only those viscometers listed in Table A1.1. Annex A1 gives further guidance.

6.1.2 *Automation Viscometers*—Automated apparatus may be used as long as they mimic the physical conditions, operations or processes of the manual apparatus they replace. Any viscometer, temperature measuring device, temperature control, temperature controlled bath or timing device incorporated in the automated apparatus shall conform to the specification for these components as stated in 6 of this test method. The automated apparatus shall be capable of determining kinematic viscosity of a certified reference material within the limits stated in 8.2.1.

6.2 *Viscometer Holders*—Use viscometer holders to enable all viscometers which have the upper meniscus directly above the lower meniscus to be suspended vertically within 1° in all directions. Those viscometers whose upper meniscus is offset from directly above the lower meniscus shall be suspended vertically within 0.3° in all directions (see Specifications D 446 and ISO 3105).

6.2.1 Viscometers shall be mounted in the constant temperature bath in the same manner as when calibrated and stated on the certificate of calibration. See Specifications D 446, see Operating Instructions in Annex A1, Annex A2, and Annex A3. For those viscometers which have Tube L (see Specifications D 446) held vertical, vertical alignment shall be confirmed by using (*1*) a holder ensured to hold Tube L vertical, or (*2*) a bubble level mounted on a rod designed to fit into Tube L, or (*3*) a plumb line suspended from the center of Tube L, or (*4*) other internal means of support provided in the constant temperature bath.

6.3 *Temperature-Controlled Bath*—Use a transparent liquid bath of sufficient depth such, that at no time during the measurement of flow time, any portion of the sample in the viscometer is less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

6.3.1 *Temperature Control*—For each series of flow time measurements, the temperature control of the bath liquid shall be such that within the range from 15 to 100°C, the temperature of the bath medium does not vary by more than ±0.02°C of the selected temperature over the length of the viscometer, or between the position of each viscometer, or at the location of the thermometer. For temperatures outside this range, the deviation from the desired temperature must not exceed± 0.05°C.

6.4 *Temperature Measuring Device in the Range from 0 to 100°C*—Use either calibrated liquid-in-glass thermometers (Annex A2) of an accuracy after correction of ±0.02°C or better, or any other thermometric device of equal or better accuracy.

6.4.1 If calibrated liquid-in-glass thermometers are used, the use of two thermometers is recommended. The two thermometers shall agree within 0.04°C.

6.4.2 Outside the range from 0 to 100°C, use either calibrated liquid-in-glass thermometers of an accuracy after correction of ± 0.05°C or better, or any other thermometric device of equal or better accuracy. When two temperature measuring devices are used in the same bath, they shall agree within ±0.1°C.

6.4.3 When using liquid-in-glass thermometers, such as those in Table A2.2, use a magnifying device to read the thermometer to the nearest ⅕ division (for example, 0.01°C or 0.02°F) to ensure that the required test temperature and temperature control capabilities are met (see 9.1). It is recommended that thermometer readings (and any corrections supplied on the certificates of calibrations for the thermometers) be recorded on a periodic basis to demonstrate compliance with the test method requirements. This information can be quite useful, especially when investigating issues or causes relating to testing accuracy and precision.

6.5 *Timing Device*—Use any timing device that is capable of taking readings with a discrimination of 0.1 s or better, and has an accuracy within ±0.07 % (see Annex A3) of the reading when tested over intervals of 200 and 900 s.

6.5.1 Electrical timing devices may be used if the current frequency is controlled to an accuracy of 0.05 % or better. Alternating currents, as provided by some public power systems, are intermittently rather than continuously controlled. When used to actuate electrical timing devices, such control can cause large errors in kinematic viscosity flow time measurements.

7. Reagents and Materials

7.1 *Chromic Acid Cleaning Solution*, or a nonchromium-containing, strongly oxidizing acid cleaning solution. (**Warning**—Chromic acid is a health hazard. It is toxic, a recognized carcinogen, highly corrosive, and potentially hazardous in contact with organic materials. If used, wear a full face-shield and full-length protective clothing including suitable gloves. Avoid breathing vapor. Dispose of used chromic acid carefully as it remains hazardous. Nonchromium-containing, strongly oxidizing acid cleaning solutions are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.)

7.2 *Sample Solvent*, completely miscible with the sample. Filter before use.

7.2.1 For most samples a volatile petroleum spirit or naphtha is suitable. For residual fuels, a prewash with an aromatic solvent such as toluene or xylene may be necessary to remove asphaltenic material.

7.3 *Drying Solvent*, a volatile solvent miscible with the sample solvent (see 7.2) and water (see 7.4). Filter before use.

7.3.1 Acetone is suitable.

7.4 *Water*, deionized or distilled and conforming to Specification D 1193 or Grade 3 of ISO 3696. Filter before use.

8. Calibration and Verification

8.1 *Viscometers*—Use only calibrated viscometers, thermometers, and timers as described in Section 6.

8.2 *Certified Viscosity Reference Standards*[8] *(Table A1.2)*—These are for use as confirmatory checks on the procedure in the laboratory.

8.2.1 If the determined kinematic viscosity does not agree within ± 0.35 % of the certified value, recheck each step in the procedure, including thermometer and viscometer calibration, to locate the source of error. Annex A1 gives details of standards available.

8.2.2 The most common sources of error are caused by particles of dust lodged in the capillary bore and temperature measurement errors. It must be appreciated that a correct result obtained on a standard oil does not preclude the possibility of a counterbalancing combination of the possible sources of error.

8.3 The calibration constant, C, is dependent upon the gravitational acceleration at the place of calibration and this must, therefore, be supplied by the standardization laboratory together with the instrument constant. Where the acceleration of gravity, g, differs by more that 0.1 %, correct the calibration constant as follows:

$$C_2 = (g_2/g_1) \times C_1 \quad (1)$$

where the subscripts 1 and 2 indicate, respectively, the standardization laboratory and the testing laboratory.

9. General Procedure for Kinematic Viscosity

9.1 Adjust and maintain the viscometer bath at the required test temperature within the limits given in 6.3.1 taking account of the conditions given in Annex A2 and of the corrections supplied on the certificates of calibration for the thermometers.

9.1.1 Thermometers shall be held in an upright position under the same conditions of immersion as when calibrated.

9.1.2 In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used (see 6.4).

9.1.3 They should be viewed with a lens assembly giving approximately five times magnification and be arranged to eliminate parallax errors.

9.2 Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time shall not be less than 200 s or the longer time noted in Specifications D 446.

9.2.1 The specific details of operation vary for the different types of viscometers listed in Table A1.1. The operating instructions for the different types of viscometers are given in Specifications D 446.

9.2.2 When the test temperature is below the dew point, fill the viscometer in the normal manner as required in 10.1. To

[8] The ASTM Viscosity Oil Standards are available in 1-pt (0.47 L) containers. Purchase orders should be addressed to the Cannon Instrument Co., P.O. Box 16, State College, PA 16804. Shipment will be made as specified or by best means.

ensure that moisture does not condense or freeze on the walls of the capillary, draw the test portion into the working capillary and timing bulb, place rubber stoppers into the tubes to hold the test portion in place, and insert the viscometer into the bath. After insertion, allow the viscometer to reach bath temperature, and the remove the stoppers. When performing manual viscosity determinations, do not use those viscometers which cannot be removed from the constant temperature bath for charging the sample portion.

9.2.2.1 The use of loosely packed drying tubes affixed to the open ends of the viscometer is permitted, but not required. If used, the drying tubes shall fit the design of the viscometer and not restrict the flow of the sample by pressures created in the instrument.

9.2.3 Viscometers used for silicone fluids, fluorocarbons, and other liquids which are difficult to remove by the use of a cleaning agent, shall be reserved for the exclusive use of those fluids except during their calibration. Subject such viscometers to calibration checks at frequent intervals. The solvent washings from these viscometers shall not be used for the cleaning of other viscometers.

10. Procedure for Transparent Liquids

10.1 Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated. If the sample contains solid particles, filter during charging through a (75-μm) filter (see Specifications D 446). If the sample is thought or known to contain fibers or solid particles, filter through a 75 μm screen, either prior to or during charging (see Specifications D 446).

NOTE 2—To minimize the potential of particles passing through the filter from aggregating, it is recommended that the time lapse between filtering and charging be kept to a minimum.

10.1.1 In general, the viscometers used for transparent liquids are of the type listed in Table A1.1, A and B.

10.1.2 With certain products which exhibit *gel-like* behavior, exercise care that flow time measurements are made at sufficiently high temperatures for such materials to flow freely, so that similar kinematic viscosity results are obtained in viscometers of different capillary diameters.

10.1.3 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Where one bath is used to accommodate several viscometers, never add or withdraw a viscometer while any other viscometer is in use for measuring a flow time.

10.1.4 Because this time will vary for different instruments, for different temperatures, and for different kinematic viscosities, establish a safe equilibrium time by trial.

10.1.4.1 Thirty minutes should be sufficient except for the highest kinematic viscosities.

10.1.5 Where the design of the viscometer requires it, adjust the volume of the sample to the mark after the sample has reached temperature equilibrium.

10.2 Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 7 mm above the first timing mark, unless any other value is stated in the operating instructions for the viscometer. With the sample flowing freely, measure, in seconds to within 0.1 s, the time required for the meniscus to pass from the first to the second timing mark. If this flow time is less than the specified minimum (see 9.2), select a viscometer with a capillary of smaller diameter and repeat the operation.

10.2.1 Repeat the procedure described in 10.2 to make a second measurement of flow time. Record both measurements.

10.2.2 If the two determinations of kinematic viscosity, calculated from the flow time measurements, agree within the stated determinability figure (see 16.1) for the product, use the average of these determinations to calculate the kinematic viscosity result to be reported. Record the result. If the determinations of kinematic viscosity do not agree within the stated determinability, repeat the measurements of flow times after thorough cleaning and drying of the viscometers and filtering (where required, see 10.1) of the sample. If the material or temperature, or both, is not listed in 16.1, for temperatures between 15 and 100°C use as an estimate of the determinability 0.20 %, and 0.35 % for temperatures outside this range.

11. Procedure for Opaque Liquids

11.1 For steam-refined cylinder oils and black lubricating oils, proceed to 11.3 ensuring a thoroughly representative sample is used. The kinematic viscosity of residual fuel oils and similar waxy products can be affected by the previous thermal history and the following procedure described in 11.1.1-11.2.2 shall be followed to minimize this.

11.1.1 In general, the viscometers used for opaque liquids are of the reverse-flow type listed in Table A1.1, C.

11.1.2 Heat in the original container, in an oven, at 60 ± 2°C for 1 h.

11.1.3 Thoroughly stir the sample with a suitable rod of sufficient length to reach the bottom of the container. Continue stirring until there is no sludge or wax adhering to the rod.

11.1.4 Recap the container tightly and shake vigorously for 1 min to complete the mixing.

11.1.4.1 With samples of a very waxy nature or oils of high kinematic viscosity, it may be necessary to increase the heating temperature above 60°C to achieve proper mixing. The sample should be sufficiently fluid for ease of stirring and shaking.

11.2 Immediately after completing 11.1.4, pour sufficient sample to fill two viscometers into a 100-mL glass flask and loosely stopper.

11.2.1 Immerse the flask in a bath of boiling water for 30 min. (**Warning**—Exercise care as vigorous boil-over can occur when opaque liquids which contain high levels of water are heated to high temperatures.)

11.2.2 Remove the flask from the bath, stopper tightly, and shake for 60 s.

11.3 Charge two viscometers in the manner dictated by the design of the instrument. For example, for the cross-arm or the BS U-tube viscometers for opaque liquids, filter the sample through a 75-μm filter into two viscometers previously placed in the bath. For samples subjected to heat treatment, use a preheated filter to prevent the sample coagulating during the filtration.

11.3.1 Viscometers which are charged before being inserted into the bath may need to be preheated in an oven prior to charging the sample. This is to ensure that the sample will not be cooled below test temperature.

11.3.2 After 10 min, adjust the volume of the sample (where the design of the viscometer requires) to coincide with the filling marks as in the viscometer specifications (see Specifications D 446).

11.3.3 Allow the charged viscometers enough time to reach the test temperature (see 11.3.1). Where one bath is used to accommodate several viscometers, never add or withdraw a viscometer while any other viscometer is in use for measuring flow time.

11.4 With the sample flowing freely, measure in seconds to within 0.1 s, the time required for the advancing ring of contact to pass from the first timing mark to the second. Record the measurement.

11.4.1 In the case of samples requiring heat treatment described in 11.1 through 11.2.1, complete the measurements of flow time within 1 h of completing 11.2.2. Record the measured flow times.

11.5 Calculate kinematie viscosity, ν, in mm ^{2}/s, from each measured flow time.

11.5.1 For residual fuel oils, if the two determinations of kinematic viscosity agree within the stated determinability figure (see 16.1), use the average of these determinations to calculate the kinematic viscosity result to be reported. Record the result. If the calculated kinematic viscosities do not agree, repeat the measurements of flow times after thorough cleaning and drying of the viscometers and filtering of the sample. If the material or temperature, or both, is not listed in 16.1, for temperatures between 15 and 100°C use as an estimate of the determinability 1.0 %, and 1.5 % for temperatures outside this range; it must he realized that these materials can be non-Newtonian, and can contain solids whieh can come out of solution as the flow time is being measured.

11.5.2 For other opaque liquids, no precision data is available.

12. Cleaning of Viscometer

12.1 Between successive determinations of kinematic viscosity, clean the viscometer thoroughly by several rinsings with the sample solvent, followed by the drying solvent (see 7.3). Dry the tube by passing a slow stream of filtered dry air through the viscometer for 2 min or until the last trace of solvent is removed.

12.2 Periodically clean the viscometer with the cleaning solution (**Warning**—see 7.1), for several hours to remove residual traces of organic deposits, rinse thoroughly with water (7.4) and drying solvent (see 7.3), and dry with filtered dry air or a vacuum line. Remove any inorganic deposits by hydrochlorie acid treatment before the use of cleaning acid, particularly if the presence of barium salts is suspected. (**Warning**—It is essential that alkaline eleaning solutions are not used as changes in the viscometer calibration can occur.)

13. Calculation

13.1 Calculate the kinematic viscosity, ν, from the measured flow time, t, and the viscometer constant, C, by means of the following equation:

$$\nu = C \cdot t \quad (2)$$

where:

ν = kinematic viscosity, mm^2/s,
C = calibration coustant of the viscometer, (mm^2/s)/s, and
t = mean flow time, s.

13.2 Calculate the dynamic viscosity, η, from the calculated kinematic viscosity, ν, and the density, ρ, by means of the following equation:

$$\eta = \nu \times \rho \times 10^{-3} \quad (3)$$

where:

η = dynamic viscosity, mPa·s,
ρ = density, kg/m^3, at the same temperature used for the determination of the kinematic viscosity, and
ν = kinematic viscosity, mm^2/s.

13.2.1 The density of the sample can be determined at the test temperature of the kinematic viscosity determination by an appropriate method such as Test Methods D 1217, D 1480, or D 1481.

14. Expression of Results

14.1 Report the test results for the kinematic or dynamie viscosity, or both, to four significant figures, together with the test temperature.

15. Report

15.1 Report the following information:

15.1.1 Type and identifieation of the product tested,

15.1.2 Reference to this test method or a corresponding international standard,

15.1.3 Result of the test (see Section 14),

15.1.4 Any deviation, by agreement or otherwise, from the procedure specified,

15.1.5 Date of the test, and

15.1.6 Name and address of the test laboratory.

16. Precision

16.1 *Determinability (d)*—The difference between successive determinations obtained by the same operator in the same laboratory using the same apparatus for a series of operations leading to a single result, would in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty:[9,10,11,12,13,14,15,16]

[9] These precision values were obtained by statistical examination of interlaboratory results from six mineral oils (base oils without additive package) in the range from 8 to 1005 mm^2/s at 40°C and from 2 to 43 mm ^{2}/s at 100°C, and were first published in 1989. Precision data available from ASTM Headquarters. Request RR:D02-1331 and RR:D02-1132. See Guide D 6074.

[10] These precision values were obtained by statistical examination of interlaboratory results from seven fully formulated engine oils in the range from 36 to 340 mm^2/s at 40°C and from 6 to 25 mm ^{2}/s at 100°C. and were first published in 1991. Precision data available from ASTM Headquarters. Request RR:D02-1332. See Guide D 6071.

Base oils at 40 and 100°C[9]	0.0020 y	(0.20 %)
Formulated oils at 40 and 100°C[10]	0.0013 y	(0.13 %)
Formulated oils at 150°C[11]	0.015 y	(1.5 %)
Petroleum wax at 100°C[12]	0.0080 y	(0.80 %)
Residual fuel oils at 80 and 100°C[13]	0.011 (y + 8)	
Residual fuel oils at 50°C[13]	0.017 y	(1.7 %)
Additives at 100°C[14]	0.00108 $y^{1.1}$	
Gas oils at 40°C[15]	0.0013 (y+1)	
Jet fuels at –20°C[16]	0.0018 y	(0.18 %)

where: y is the average of determinations being compared.

16.2 *Repeatability (r)*—The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated only in one case in twenty:[9,10,11,12,13,14,15,16]

Base oils at 40 and 100°C[9]	0.0011 x	(0.11 %)
Formulated oils at 40 and 100°C[10]	0.0026 x	(0.26 %)
Formulated oils at 150°C[11]	0.0056 x	(0.56 %)
Petroleum wax at 100°C[12]	0.0141 $x^{1.2}$	
Residual fuel oils at 80 and 100°C[13]	0.013 (x + 8)	
Residual oils at 50°C[13]	0.015 x	(1.5 %)
Additives at 100°C[14]	0.00192 $x^{1.1}$	
Gas oils at 40°C[15]	0.0043 (x+1)	
Jet fuels at –20°C[16]	0.007 x	(0.7 %)

where: x is the average of results being compared.

16.3 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated below only in one case in twenty.[9,10,11,12,13,14,15,16]

Base oils at 40 and 100°C[9]	0.0065 x	(0.65 %)
Formulated oils at 40 and 100°C	0.0076 x	(0.76 %)
Formulated oils at 150°C[11]	0.018 x	(1.8 %)
Petroleum wax at 100°C[12]	0.0366 $x^{1.2}$	
Residual fuel oils at 80 and 100°C[13]	0.04 (x + 8)	
Residual oils at 50°C[13]	0.074 x	(7.4 %)
Additives at 100°C[14]	0.00862 $x^{1.1}$	
Gas oils at 40°C[15]	0.0082 (x+1)	
Jet fuels at –20°C[16]	0.019 x	(1.9 %)

where: x is the average of results being compared.

16.4 The precision for used oils has not been determined but is expected to be poorer than that for formulated oils. Because of the extreme variability of such used oils, it is not anticipated that the precision of used oils will be determined.

16.5 The precision for specific automated viscometers has not been determined. However, an analysis has been made of a large data set including both automated and manual viscometers over the temperature range of 40 to 100°C. The reproducibility of automated viscometer data is not statistically significantly different than the reproducibility of manual viscometer data. It is also shown that there is no bias of the automated data in comparison to the manual data.[17]

17. Keywords

17.1 dynamic viscosity; kinematic viscosity; viscometer; viscosity

[11] These precision values were obtained by statistical examination of interlaboratory results for eight fully formulated engine oils in the range from 7 to 19 mm ^{2}/s at 150°C, and first published in 1991. Precision data available from ASTM Headquarters. Request RR:D02-1333. See Guide D 6074.

[12] These precision values were obtained by statistical examination of interlaboratory results from five petroleum waxes in the range from 3 to 16 mm^2/s at 100°C, and were first published in 1988. Precision data available from ASTM Headquarters. Request RR:D02-1334.

[13] These precision values were obtained by statistical examination of interlaboratory results from fourteen residual fuel oils in the range from 30 to 1300 mm ^{2}/s at 50°C and from 5 to 170 mm^2/s at 80 and 100°C, and were first published in 1984. Precision data available from ASTM Headquarters. Request RR:D02-1198.

[14] These precision values were obtained by statistical examination of interlaboratory results from eight additives in the range from 145 to 1500 mm^2/s at 100°C and were first available in 1997. Precision data available from ASTM Headquarters. Request RR:D02-1421.

[15] These precision values were obtained by statistical examination of interlaboratory results from eight gas oils in the range from 1 to 13 mm^2/s at 40°C and were first available in 1997. Precision data available from ASTM Headquarters. Request RR:D02-1422.

[16] These precision values were obtained by statistical examination of interlaboratory results from nine jet fuels in the range from 4.3 to 5.6 mm ^{2}/s at– 20°C and were first available in 1997. Precision data available from ASTM Headquarters. Request RR:D02-1420.

[17] Precision data available from ASTM Headquarters. Request RR: D02–1498.

ANNEXES

(Mandatory Information)

A1. VISCOMETER TYPES, CALIBRATION, AND VERIFICATION

A1.1 Viscometer Types

A1.1.1 Table A1.1 lists capillary viscometers commonly in use for viscosity determinations on petroleum products. For specifications and operating instructions, refer to Specifications D 446.

A1.2 Calibration

A1.2.1 Calibrate working standard viscometers against master viscometers having a certificate of calibration traceable to a national standard. Viscometers used for analysis shall be calibrated in comparison with working standard viscometers or master viscometers, or by the procedures given in Specifications D 446 or ISO 3105. Viscometer constants shall be measured and expressed to the nearest 0.1 % of their value.

A1.3 Verification

A1.3.1 Viscometer constants shall either be verified by a similar procedure to A1.2, or conveniently checked by means of certified viscosity oils.

A1.3.2 These oils can be used for confirmatory checks on the procedure in a laboratory. If the measured viscosity does not agree within ± 0.35 % of the certified value, recheck each

TABLE A1.1 Viscometer Types

Viscometer Identification	Kinematic Viscosity Range,[A] mm^2/s
A. Ostwald Types for Transparent Liquids	
Cannon-Fenske routine[B]	0.5 to 20 000
Zeitfuchs	0.6 to 3 000
BS/U-tube[B]	0.9 to 10 000
BS/U/M miniature	0.2 to 100
SIL[B]	0.6 to 10 000
Cannon-Manning semi-micro	0.4 to 20 000
Pinkevitch[B]	0.6 to 17 000
B. Suspended-level Types for Transparent Liquids	
BS/IP/SL[B]	3.5 to 100 000
BS/IP/SL(S)[B]	1.05 to 10 000
BS/IP/MSL	0.6 to 3 000
Ubbelohde[B]	0.3 to 100 000
FitzSimons	0.6 to 1 200
Atlantic[B]	0.75 to 5 000
Cannon-Ubbelohde(A), Cannon Ubbelohde dilution[B](B)	0.5 to 100 000
Cannon-Ubbelohde semi-micro	0.4 to 20 000
C. Reverse-flow Types for Transparent and Opaque Liquids	
Cannon-Fenske opaque	0.4 to 20 000
Zeitfuchs cross-arm	0.6 to 100 000
BS/IP/RF U-tube reverse-flow	0.6 to 300 000
Lantz-Zeitfuchs type reverse-flow	60 to 100 000

[A] Each range quoted requires a series of viscometers. To avoid the necessity of making a kinetic energy correction, these viscometers are designed for a flow time in excess of 200 s except where noted in Specifications D 446.

[B] In each of these series, the minimum flow time for the viscometers with lowest constants exceeds 200 s.

step in the procedure including thermometer, timer, and viscometer calibration to loeate the source of error. It should be appreciated that a correct result obtained on a certified oil does not preclude the possibility of a counterbalancing combination of thc possible sources of error.

A1.3.2.1 A range of viscosity oil standards is commercially available, and each oil carries a certification of the measured value established by multiple testing. Table A1.2 gives the standard range of oils, together with the approximate viscosities over a range of temperatures.

TABLE A1.2 Viscosity Oil Standards[A]

Designation	Approximate Kinematic Viscosity, mm^2/s					
	–40°C	20°C	25°C	40°C	50°C	100°C
S3	80	4.6	4.0	2.9	...	1.2
S6	...	11	8.9	5.7	...	1.8
S20	...	44	34	18	...	3.9
S60	...	170	120	54	...	7.2
S200	...	640	450	180	...	17
S600	...	2 400	1 600	520	280	32
S2 000	...	8 700	5 600	1 700	...	75
S8 000	...	37 000	23 000	6 700	...	...
S30 000	...	...	81 000	23 000	11 000	...

[A] The actual values for these standards are established and annually reaffirmed by cooperative tests. In 1991, tests were made using 15 different types of viscometers in 28 laboratories located in 14 countries.

[B] Kinematic viscosities may also be supplied at 100°F.

[C] Kinematic viscosities may also be supplied at 210°F.

S-3

Designation: D 287 – 92 (Reapproved 2000)$^{\epsilon 1}$

An American National Standard

Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)[1]

This standard is issued under the fixed designation D 287: the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. This test method has been adopted for use by government agencies to replace Method 401 of Federal Test Method Standard No. 791b.

ϵ^1 NOTE—Editorial corrections were made throughout in April 2000.

1. Scope

1.1 This test method covers the determination by means of a glass hydrometer of the API gravity of crude petroleum and petroleum products normally handled as liquids and having a Reid vapor pressure (Test Method D 323) of 26 psi (180 kPa) or less. Gravities are determined at 60°F (15.56°C), or converted to values at 60°F, by means of standard tables. These tables are not applicable to nonhydrocarbons or essentially pure hydrocarbons such as the aromatics.

NOTE 1—The international version of this test method is described in Practice D 1298.

1.2 Values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are provided for information purposes only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see 8.3.

2. Referenced Documents

2.1 *ASTM Standards:*

D 323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)[2]

D 1250 Guide for Petroleum Measurement Tables[2]

D 1298 Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method[2]

E 1 Specification for ASTM Thermometers[3]

E 100 Specification for ASTM Hydrometers[3]

2.2 *IP Standards:*[4]

Specifications for IP Standard Thermometers

IP Specifications for Petroleum Hydrometers

3. Terminology

3.1 *Definitions:*

3.1.1 *API gravity*—a special function of relative density (specific gravity) 60/60°F (15.56/15.56°C), represented by:

$$\text{API gravity, deg} = (141.5/\text{sp gr } 60/60°\text{F}) - 131.5 \quad (1)$$

No statement of reference temperature is required, since 60°F is included in the definition.

4. Summary of Test Method

4.1 This test method is based on the principle that the gravity of a liquid varies directly with the depth of immersion of a body floating in it. The floating body, which is graduated by API gravity units in this method, is called an API hydrometer.

4.2 The API gravity is read by observing the freely floating API hydrometer and noting the graduation nearest to the apparent intersection of the horizontal plane surface of the liquid with the vertical scale of the hydrometer, after temperature equilibrium has been reached. The temperature of the sample is read from a separate accurate ASTM thermometer in the sample or from the thermometer which is an integral part of the hydrometer (thermohydrometer).

5. Significance and Use

5.1 Accurate determination of the gravity of petroleum and its products is necessary for the conversion of measured volumes to volumes at the standard temperature of 60°F (15.56°C).

5.2 Gravity is a factor governing the quality of crude oils. However, the gravity of a petroleum product is an uncertain

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.02.0A on Temperature, Density, Physical Properties.

Current edition approved Aug. 15, 1992. Published October 1992. Originally published as D 287–28T, replacing former D 47. Last previous edition D 287–82 (1987)$^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

[4] Available from the Institute of Petroleum, 61 New Cavendish St., London WIM. 8AR. England.

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indication of its quality. Correlated with other properties, gravity can be used to give approximate hydrocarbon composition and heat of combustion.

6. Apparatus

6.1 *Hydrometers*, of glass, graduated in degrees API as listed in Table 1 and conforming to Specification E 100, or the IP Specifications for Petroleum Hydrometers.

6.2 *Thermometers*, having a range from – 5 to + 215°F and conforming to the requirements for Thermometer 12F as prescribed in Specification E 1 or Thermometer 64F of the Specifications for IP Standard Thermometers. A thermometer is not required if a thermohydrometer is employed.

NOTE 2—The ASTM Gravity Thermometer 12F has 0.5°F subdivisions and allowable ±0.25°F scale error. The thermometers incorporated in thermohydrometers have 2°F subdivisions and allowable ±1°F scale error.

6.3 *Hydrometer Cylinders*, of metal, clear glass, or plastic. For convenience in pouring, the cylinder may have a lip on the rim. The inside diameter of the cylinder shall be at least 25 mm greater than the outside diameter of the hydrometer used in it. The height of the cylinder shall be such that the length of the column of sample it contains is greater by at least 25 mm than the portion of the hydrometer which is immersed beneath the surface of the sample. For field testing, a sampling thief of suitable dimensions may be used.

7. Temperature of Test

7.1 The gravity determined by the hydrometer method is most accurate at or near the standard temperature of 60°F (15.56°C). Use this or any other temperature between 0 and 195°F (–18 and + 90°C) for the test, so far as it is consistent with the type of sample and necessary limiting conditions shown in Table 2.

8. Procedure

8.1 For referee testing, use the long plain form of hydrometer (1H to 10H). For field testing, use the thermohydrometer.

8.2 Adjust the temperature of the sample in accordance with Table 2. For field testing, test temperatures other than those listed in Table 2 may be used. The hydrometer cylinder shall be approximately the same temperature as the sample to be tested.

8.3 Transfer the sample into the clean hydrometer cylinder without splashing, so as to avoid the formation of air bubbles and to reduce to a minimum the evaporation of the lower boiling constituents of the more volatile samples. (**Warning**—Extremely flammable. Vapors may cause flash fire.) For the more volatile samples, transfer to the hydrometer cylinder by siphoning. (**Warning**—Do not start the siphon by mouth.) Use a rubber aspirator bulb. Remove any air bubbles formed, after they have collected on the surface of the sample, by touching them with a piece of clean filter paper before inserting the hydrometer. For field testing, make the gravity measurement directly in the sampling thief. Place the cylinder containing the sample in a vertical position in a location free from air currents. Take precautions to prevent the temperature of the sample from changing appreciably during the time necessary to complete the test. During this period, the temperature of the surrounding medium should not change more than 5°F (2°C).

TABLE 1 Available Hydrometers Scaled, Degrees API

Designation	Type	API Range, deg		Scale	
		Series Total	Each Unit	Division	Error
1H to 10H	long plain	–1 to 101	12	0.1	0.1
21H to 40H	short plain	0 to 101	6	0.1	0.2
51H to 60H	thermo	–1 to 101	12	0.1	0.1
71H to 74H	thermo	–1 to 41	12	0.1	0.1
[A]	thermo	15 to 51	8		

[A] Eight-degree range thermohydrometers are available.

8.4 Lower the hydrometer gently into the sample and, when it has settled, depress it about two scale divisions into the liquid and then release it; keep the rest of the stem dry, as unnecessary liquid on the stem changes the effective weight of the instrument, and so affects the reading obtained. With samples of low viscosity, a slight spin imparted to the instrument on releasing assists in bringing it to rest, floating freely away from the walls of the hydrometer cylinder. Allow sufficient time for the hydrometer to become completely stationary and for all air bubbles to come to the surface. This is particularly necessary in the case of the more viscous samples.

8.5 When the hydrometer has come to rest, floating freely, and the temperature of the sample is constant to 0.2°F (0.1°C), read the hydrometer to the nearest scale division. The correct reading is that point on the hydrometer scale at which the surface of the liquid cuts the scale. Determine this point by placing the eye slightly below the level of the liquid and slowly raising it until the surface, first seen as a distorted ellipse, appears to become a straight line cutting the hydrometer scale.

8.6 To make a reading with nontransparent liquids, observe the point on the hydrometer scale to which the sample rises above its main surface, placing the eye slightly above the plane surface of the liquid. This reading requires a correction. Determine this correction for the particular hydrometer in use by observing the height above the main surface of the liquid to which the sample rises on the hydrometer scale when the hydrometer in question is immersed in a transparent liquid having a surface tension similar to that of a sample under test.

8.7 Observe the temperature of the sample to the nearest 0.25°F (0.1°C) immediately before and after the observation of the gravity, the liquid in the cylinder being thoroughly but cautiously stirred with the thermometer (Note 3), and the whole of the mercury thread being immersed. Should these temperature readings differ by more than 1°F (0.5°C), repeat the temperature and gravity observations when the temperature of the sample has become more stable. Record the mean of the thermometer reading before and after the final hydrometer reading, to the nearest 1°F, as the temperature of the test.

NOTE 3—When thermohydrometers are used, stir the sample by carefully raising and lowering the hydrometer. It is satisfactory in this case to read the thermometer scale after the hydrometer reading has been observed. Read the thermometer to the nearest 1°F (0.5°C).

9. Calculation

9.1 When gravities have been observed on opaque liquids using the procedure given in 8.6, subtract the correction from the hydrometer reading observed.

9.2 Correct all hydrometer readings to 60°F (15.56°C), using Tables 5A or Tables 5B of Guide D 1250.

TABLE 2 Limiting Conditions and Testing Temperatures

Sample Type	Gravity Limits	Initial Boiling Point Limits	Other Limits	Test Temperature
Highly volatile	lighter than 70° API			Cool to 35°F (2°C) or lower in original closed container.
Moderately volatile	heavier than 70° API	below 250°F (120°C)		Cool to 65°F (18°C) or lower in original closed container.
Moderately volatile and viscous	heavier than 70° API	below 250°F (120°C)	Viscosity too high at 65°F (18°C)	Heat to minimum temperature for sufficient fluidity.
Nonvolatile	heavier than 70° API	above 250°F (120°C)		Any temperature between 0 and 195°F (−18 and 90°C) as convenient.
Mixtures of nonpetroleum products or essentially pure hydrocarbons				60 ± 0.25°F (15.56 ± 0.1°C)

10. Report

10.1 Report the corrected hydrometer reading as degrees API (°API) or as API Gravity.

11. Precision and Bias

11.1 The precision of this test method as obtained by statistical examination of interlaboratory test results is as follows:

11.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed 0.2° API only in one case in twenty.

11.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators, working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed 0.5° API only in one case in twenty.

NOTE 4—The precision for this method was not obtained in accordance with RR:D02-1007.[3]

NOTE 5—This precision statement applies only to measurements made at temperatures differing from 60°F (15.56°C) by less than 18°F (10°C).

11.2 *Bias*—Bias for this method has not been determined.

12. Keywords

12.1 API gravity; crude petroleum; hydrometer; thermohydrometer; thermometer

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Attachment 1-6
Laboratory Policies and Guidelines

WATS Laboratory Policies & Guidelines

February, 2007

Weyerhaeuser Analysis and Testing Services (WATS)
Laboratory Policies and Guidelines
Table of Contents

Chapter	Topic	Page
-1 -	Quality Policy Statement.	3
-2 -	Introduction	4
-3 -	Laboratory Facilities and Environment	6
-4 -	Sample Management	8
-5 -	Determining Test Methods and Analysis	12
-6 -	Subcontracting Samples	13
-7 -	WATS Testing	14
-8 -	Equipment and Instrumentation	15
-9 -	Chemical Standards and Reagents	19
10 -	Quality Control Testing Procedures	20
11 -	Quality Control Monitoring by Control Charts	24
12 -	Detection and Quantitation Limits	25
13 -	Rounding	26
14 -	Data Recording	30
15 -	Laboratory Reports	32
16 -	Acronyms, Glossary, & Qualifiers	34

Weyerhaeuser Analysis and Testing Services (WATS) is registered to ISO 9001:2000 standards. The registration applies to the provision of pulp and paper analytical and physical laboratory services. The policies and practices delineated in this document are fully supported by the management and staff members of Weyerhaeuser Analysis and Testing Services (WATS). These policies and practices are to be adhered to by everyone working in WATS.

Revision History

WATS Quality Assurance Manual

December 13, 2004 Rewritten date. Draft proposal presented for a rewrite.

The original quality document *A&T QUALITY ASSURANCE GUIDELINES AND POLICY* was totally revised and two documents were created, *WATS Quality Assurance Manual* and *WATS Laboratory Policies and Guidelines*.

July 14, 2005 Effective date for rewritten quality manual.

Weyerhaeuser Analysis and Testing Services *WATS Quality Assurance Manual* was proposed on December 13, 2004 and became effective July 14, 2005.

October 31, 2006 Effective date for revision of quality manual.

An extensive review and rewrite of the *WATS Quality Assurance Manual* was undertaken in December, 2005 to more accurately reflect the processes and policies of WATS.

WATS Laboratory Guidelines and Policies

July, 2005 *WATS Laboratory Policies and Guidelines* issued

12-08-05 Index corrected 12-20-05 Reissued with chapter three typo corrected.

03-06-2006 Comprehensive, department wide, LP&G review being conducted by technical specialists.

February, 2007 Revised LP&G issued for review.

April, 2007 LP&G with February, 2007 revision date published.

Quality Policy Statement

1.1 WATS (Weyerhaeuser Analysis and Testing Services) emphasizes a total quality management process to provide competitive technical services in a safe, timely, accurate, ethical, and cost efficient manner. WATS sets yearly quality objectives to improve the effectiveness of the quality management system (QMS), striving to ensure individual, team, and department commitment.

1.2 WATS quality management system endeavors to give the highest degree of confidence in our data to both internal and external customers to achieve customer satisfaction at all times. We each take personal responsibility to provide a quality product while meeting the company's high standards of integrity and ethics.

1.3 WATS provides a work environment that ensures employees accessibility to all levels of management and encourages questions and expressions of concern on quality issues. We are committed to continually upgrading the skills of technical and administrative staff through formal training.

1.4 WATS is committed to compliance with applicable agency regulations and the ISO 9001:2000 Standard for its Pulp and Paper testing through quality guidelines that include the “WATS Quality Manual” and “WATS Laboratory Policy” and Guidelines.

Introduction

2.1 Weyerhaeuser Analysis and Testing Services (WATS) recognizes that the reliability and accuracy of its testing and analysis are affected by a variety of factors including:

- human factors
- accommodations
- environmental conditions
- test and calibration procedures
- test validations
- equipment
- traceability of measurements
- sampling
- handling of tests items
- equipment

2.2 WATS quality management system (QMS) strives to give the highest degree of confidence in our data to both internal and external customers to insure that customer satisfaction is achieved. "WATS Laboratory Policies and Guidelines" covers both qualitative and quantitative analyses carried out on a routine or non-routine basis.

2.3 WATS uses a Total Quality Management (TQM) approach to quality. Emphasis is on continuous improvement and customer satisfaction. WATS team members take personal responsibility to provide a quality product. Yearly quality objectives ensure individual, team, and department commitment to the quality management system (QMS). The laboratories that provide pulp and paper testing are registered to the ISO 9001:2000 standard. Customers that are required to comply with state or federal regulations are assured of the technical competence of the laboratory through round robin testing with nationally accredited laboratories. "WATS Quality Manual", "WATS Laboratory Policies and Guidelines", test methods, standard operating procedures, training records, and other records are maintained to insure quality and confidence in our product, data.

2.4 WATS facilities ensure the safekeeping of samples, reference materials, and records. WATS has appropriate equipment for carrying out the work of analysis, both chemical and physical. Refrigerators/freezers are available for the storage of samples prone to degradation. Chemical fume hoods are provided and maintained. Analysis is carried out by trained staff. Measures are taken to ensure good housekeeping in the laboratories. These processes are documented in the "WATS Quality Manual", "WATS Laboratory Policies and Guidelines", test methods, standard operating procedures, training records, and other documents.

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2.5 These guidelines extend to any temporary or mobile facility operated by WATS for mandated or regulatory environmental work. It does not apply to WATS personnel on loan to other organizations external to WATS management.

Laboratory Facilities and Environment

3.1 WATS occupies 3300 square meters (35,700 square feet) in several laboratory modules in the Weyerhaeuser Technology Center on the Weyerhaeuser Campus at 32901 Weyerhaeuser Way South, Federal Way. The facility is twenty minutes away from a major airport and has direct access to the area's major freeways. Federal Way's fire and emergency services are minutes away.

3.2 The technology center was built in 1978. It is a modern building with an up-to-date electrical system (with back-up generators), water, sufficient lighting for laboratory work, central high-purity RO water, air conditioning, central vacuum systems, and natural gas in rooms requiring it. The design of the laboratories supports good laboratory practices. Equipment in the building is monitored 24 hours a day by a Control Center that monitors room temperature, humidity, electricity, etc.

3.3 Samples, reagents, measurement standards and reference materials are stored to ensure their integrity. Samples are stored in such a way that cross contamination is not possible. The laboratories guard against their deterioration, contamination and loss of identity through the "Chemical Hygiene Plan" and related standard operating procedures (SOPS).

3.4 The environment in the laboratories where tests are performed is maintained so that conditions do not invalidate results nor adversely affect the accuracy or cause uncertainty in the measurement. Measurements are not made if environmental conditions deviate from those stated in the SOPS. A breakdown of critical environmental conditions may be indicated either by monitoring systems or by the analytical quality control within the particular tests. The impact of such failures may be assessed as part of ruggedness testing during method validation and where appropriate, emergency procedures established. Decontamination procedures may be appropriate where environment or equipment is subject to change of use or where accidental contamination has occurred.

3.5 Alarms are triggered in the Control Center when conditions are not within specifications. Follow-up procedures for alarm events are located in the Control Center's standard operating procedures. Telephone numbers are available in order to contact key personnel in emergencies. The laboratory documents deviations, generating corrective actions when environmental conditions are not within conditions specified by the laboratory. (See the standard operating procedures for nonconformances, complaints, and corrective actions.) Monitoring devices are periodically verified against accurate and traceable standards, but are not used to apply corrections that affect the quality of measurements.

3.6 It may be necessary to restrict access to particular areas of a laboratory because of the nature of the work carried out there. Restrictions might be made because of

security, safety, or sensitivity to contamination or interferences. Typical examples might be work involving explosives, radioactive materials, carcinogens, and trace analysis. Where such restrictions are in force, staff are made aware of the intended use of a particular area, the restrictions imposed on working within such areas, the reasons for imposing such restrictions, and the procedures to follow when such restrictions are breached. When selecting designated areas for new work, account must be taken of the previous use of the area. Before use, checks are made to ensure that the area is free of contamination.

3.7 Facilities and housekeeping services work with staff to insure that laboratory areas are maintained in a clean and organized manner to facilitate proper performance of calibrations and tests. Working safely is the main goal of all Weyerhaeuser's activities. Sufficient space is allocated to minimize the risk of injury to staff and/or damage to standards or equipment due to activities around test setup. A convenient and efficient work environment with effective separation of incompatible activities is maintained. Documents, policies, and procedures to work safely in a clean organized facility are found in the "WATS Quality Assurance Manual", "Chemical Hygiene Plan", standard operating procedures, test methods, and "Outsourced Services" documents.

Sample Management

4.1 Sample Submittal

4.1.1 The Sample Management Office (SMO) is responsible for receiving, documenting, and distributing samples to the laboratory staff. APG Round Robin samples, that occur twice a year, are submitted to SMO as blind and reference samples for WATS analysis.

4.1.2 Processes and procedures for the Sample Management Office (SMO) are described in OP S-SMO , except for Microstructure's SMO. There is a unique situation in the Microstructure area, and not all criteria may apply to samples submitted to them.

4.2 Sample Acceptance Policy

4.2.1 SMO, working with the Project Managers, reviews all new work to ensure that the tests selected are appropriate for the intended purpose. This review ensures that the appropriate resources are available before work begins.

4.2.2 Submitted samples not meeting the following criteria must have a nonconformance/corrective action form filled out that clearly defines the nature and substance of the variation. (The information is also noted on the report with the data, an attached narrative, or by attaching a copy of the nonconformance/corrective action form.)

- Adequate amount of sample to perform the necessary tests.
- Use of appropriate sample containers.
- Samples that show no signs of damage or contamination.
- Proper sample labeling, including unique identification. Labels should be durable and use indelible ink.
- Adherence to specified holding times.
- For regulatory samples, documentation should include sample identification, the location, date and time of collection, preservation, and any special remarks concerning the sample. The collector's name and sample type are recommended.

4.3 Sampling and Sample Handling

4.3.1 Precision and accuracy of analytical results are strongly affected by sample handling. Improper sampling preservation techniques, containers, or exceeding holding times can cause significant errors. Analysts are required to work closely with the Sample Management Team to assure the appropriate information is included with the sample. Factors that must be taken into account to ensure sound analytical results include:

- Selection of a representative sampling site
- Use of proper techniques for sampling
- Amount of sample to be taken
- Number of samples necessary to provide proper information
- The type and number of containers used for sampling
- Type of sample preservation to be used (if any)
- Proper transportation to the laboratory
- Sample storage time
- Complete documentation, including proper chain of custody

4.3.2 WATS has documented instructions on the handling and preparation of samples required for periodic compliance audits. Performance audits to monitor the quality of the laboratory's testing activities may require re-testing of retained samples. SMO's policy and procedures for managing samples submitted to WATS help ensure the quality of results.

4.4 Sample Tracking

4.4.1 The laboratory has a documented system for uniquely identifying the items to be tested, to ensure that there can be no confusion regarding the identity of such items at any time, either physically or within any records. This system includes identification for all samples, lab-generated samples, sub-samples, and subsequent extracts and/or digestates. Any knowledge of sample problems must be documented through a nonconformance form and communicated to management and the client (if appropriate).

- The laboratory shall assign a unique identification (ID) code to each sample received the laboratory.
- This laboratory code (identity) shall maintain an unequivocal link with the unique customer or field ID code assigned each 'container'.
- The laboratory ID code shall be placed on the sample or sample container as a durable label.
- The laboratory ID code shall be entered into the laboratory records and shall be the link that associates the sample with related laboratory activities such as sample preparation or calibration.

4.5 Sample Receipt Protocols

4.5.1 The condition of the sample upon receipt is recorded. This description will include describing abnormalities or departures from the standard condition as prescribed in the relevant test method. If the sample is not suitable for testing; does not conform to the description provided, or if the tests required are not fully specified, then SMO or the Project Manager will consult with the client for further instructions before proceeding. SMO determines if the sample has received all

necessary preparation. The customer may require preparation to be undertaken or arranged through SMO.

4.5.2 If a sample does not meet the "Sample Receipt Protocol", SMO either retains correspondence and/or records of conversations concerning the final disposition of rejected samples or documents any decision to proceed with the analysis of samples not meeting acceptance criteria. The condition of these samples will, at a minimum, be noted on the chain of custody or transmittal form (if present) and laboratory receipt documents and the test data shall be appropriately "qualified" on the final report.

4.5.3 All samples having temperature requirements shall be considered acceptable if the temperature upon arrival is either within 2 ºC of the required temperature or within the method specified range. For samples with a specified temperature of 4º C, samples with a temperature of 0.1 to 6º C shall be acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet these criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

4.5.4 SMO checks any applicable chemical preservation using readily available techniques, such as pH, free chlorine, or other necessary tests, prior to or during sample preparation or analysis. The results of all checks shall be recorded.

4.5.5 There is a permanent, sequential log maintained to document receipt of all sample(s) (containers). The following information must be recorded in the laboratory chronological log:

a. Date and time of laboratory receipt of sample.
b. Unique laboratory ID code.
c. ID (code) supplied by sample submitter.
d. Requested analyses, including method identification, if applicable.
e. Comments resulting from inspection for sample acceptance/rejection.
f. Signature or initials of data logger.
g. Sample collection date, if known, for environmental regulatory samples.

4.5.6 All documentation sent by the customer is retained. The complete chain-of-custody record, if utilized, shall be maintained.

4.6 Sample Custody Log

4.6.1 Upon creation of a SR, a custody log will be established for all samples. This will be used to log the location/storage condition of the sample while in the laboratory's possession. Final disposition (trash, used up in testing, hazardous waste, returned to client, etc.) of the samples shall be noted on this log.

4.7 Sample Disposal

4.7.1 Sample disposal is recorded in the Sample Custody Log. Non-chemical-like samples (mainly pulp and paper) are disposed of in normal trash. If samples are wet, free liquids are removed. Samples chemical in nature (mainly analytical) follow the laboratory disposal procedures for samples, digestates, leachates and extracts, or other sample preparation products and are specified in Appendix B-Waste Disposal Guidelines of the Chemical Hygiene Plan.

References
OP S-SMO Effective Date: 05 SEPT 2006
Chemical Hygiene Plan

Determining Test Methods and Analysis

5.1 Test methods used are based on the customer's request. Determining if these test methods are appropriate is done by evaluating the end-use needs and understanding restrictions and limitations inherent in the sample. If the method requested is not appropriate, the customer will be informed and suggestions offered. The laboratory does not override the customer's request unless there is an issue of safety.

5.2 Regulations identify specific methods for certain tests. NPDES permit-testing, RCRA regulations, SDWA, and FDA all have legal requirements or recognized standard requirements for testing. These must be followed for compliance with product specifications or to assure legal defensibility in civil cases.

5.3 Accuracy and precision are the major criteria in selecting a test method. The method must generate data that meets the customer's needs. Determining the appropriate test method to use to analyze samples requires paying attention to possible sources of error in the analysis. The more complex a procedure is, the less reliable the results will be. Reagent purity and stability, instrument variability, and method complexity affect the reliability of the procedure.

5.4 Procedures should be compatible with the matrix being measured. Paper may not respond in a test method in the same manner that pulp would. Pulp or paper procedures may not be appropriate for plastic impregnated paper or linerboard. Total metal (acid leach) may not be appropriate for total metals on a scale sample (HF digest to give silicates) or metals in pulp.

5.6 The limit of detection of the method will determine if the method is sensitive to the analyte at the concentration of interest. Keep the limit of detection in mind when sampling as it can affect sampling procedures.

5.7 The sample matrix can affect the choice of methods. Possible contaminants can interfere with an analysis. It may be necessary to determine if a method will be affected by any suspected contaminants. A method should be chosen that generates a proper response of the analyte of interest.

5.8 Historical requirements may make it necessary to run a specific test method. It may be necessary to generate information to compare to previous data. If the method requested is not appropriate, then the customer will be informed and suggestions may be offered. The laboratory does not override the customer's request unless there is an issue of safety.

Subcontracting Samples

6.1 If samples need to be subcontracted, clients should be encouraged to submit their samples directly to an outside organization rather than passing them through WATS' laboratories.

6.2 There are established procedures for the review of client requests (contracts). The review of a request (contract) must include reviewing any work to be subcontracted by the laboratory. The client must be notified of any deviation from the contract. Differences between the SR and the client's request must be resolved before any work begins. If the work has started, and the SR needs to be amended, the review process must be followed again. The Service Request (SR) is the laboratory's documentation for most client requests. The request may be oral; however, the conversation must be noted on the SR.

6.3 WATS will advise the client if it is going to subcontract any portion of the testing to another organization.

6.4 When subcontracting, work will be placed with a laboratory that can meet the needs of the client. The project manager will evaluate the quality of the laboratory's work or determine if the laboratory has appropriate accreditation and/or certification for the tests to be performed.

6.5 Subcontracted work appearing on WATS generated reports will be clearly identified as subcontracted work.

WATS Testing

7.1 WATS uses appropriate up-to-date procedures for all tests and related activities within its responsibility and scope of accreditation. All ISO or regulatory procedures are performed following documented test methods. Methods required for regulatory work will conform to published national or international methods when necessary.

7.2 Weyerhaeuser's own documents for standard operating procedures, test methods, and calibration procedures are used to assure the repeatability and validity of the tests done and the analysis provided. These test methods and procedures help assure consistency and help to assess sources of error by following a standard format detailed in "Methods and SOP Writing Process", OQ O-Proc Wrt. If an analysis does not follow the recognized Weyerhaeuser test method, many of which are adaptations of nationally published, and internationally recognized procedures (as referenced in the published method), every reasonable effort will be made to inform the customer and the final report will note the modification. Nonconformances attached to the SR will document any deviations.

7.3 If the customer requests a method that is not appropriate, the customer will be informed and suggestions will be offered. Some regulations require following methods that may have had many subsequent revisions. The lab must comply with the regulations.

7.4 Custom testing or non-standard testing requests must be documented and be in agreement with the customer's end-use needs. A copy must be kept with the Service Request (SR). A procedure for the review of customer requests (contracts) is located in the standard operating procedure for the Sample Management Organization (SMO) department. The request may be oral. The SR is SMO's documentation for customer requests. Differences between the SR and the customer's request must be resolved before work begins.

Equipment, Instrumentation, Standards and Associated Apparatus

8.1 All measuring operations, testing equipment and reference standards having an effect on the accuracy and validity of tests shall be calibrated before being placed into service and verified on a continuing base. If WATS requires the use of equipment outside its permanent control, it shall ensure that the relevant requirements of the "WATS Quality Manual" and "Laboratory Policies and Guidelines" are met. WATS will not be responsible for calibration and standardization for equipment on loan.

8.1.1 Documentation

8.1.2 A WATS's spreadsheet (administrative maintained) is used to identify the equipment (by a unique ID), including name; manufacture; model and type; serial number; date received; and date placed in service. This spreadsheet is also used (if applicable) to track renewal dates and to provide reminders (to relevant technical staff) when calibration and preventative maintenance are due (with a frequency of at least 1 year for most equipment).

8.1.3 Instrument lab books are maintained on any data generating and monitoring equipment and on all reference materials significant to the test performed. These records include documentation on all routine (calibration and reference material verification) and non-routine (repair) maintenance activities. The information must include who did the maintenance; the date of maintenance; the condition 'as left' and 'as found'; a description of any repair; and identification of parts replaced.

8.1.4 The instrument logbooks are either located near the instruments or in electronic format on the computer that controls the instrument. Monitoring equipment (balances, ovens, refrigerators, freezers, and water distillation and Millipore systems) logbooks are located with the personnel responsible for checking the applicable equipment. Maintenance receipts are filed with the administrative staff.

8.1.5 All instruction and maintenance manuals (supplied by the manufacturer), reference data (relevant to the laboratory work) and instructions (WATS SOPs) shall be maintained and readily available to the staff. These are located near the instrument (or in a central location, such as a file cabinet or Panagon).

8.1.6 Courses, workshops, and manufacturer's training sessions on specific equipment shall be part of the employee's training records. It is the responsibility of the employee to document the training and submit the documentation to the administrative personnel. They store the information.

8.2 Equipment and Instrument Maintenance

8.2.1 Equipment and instruments must be kept in good working order. A comprehensive, preventative maintenance program is followed to maintain proper performance, to prevent its failure during use and to increase the reliability of the measurements. The degree of the preventative maintenance relies on the manufacturer's recommendations and previous documented experience with the equipment. Where appropriate, spare parts should be available in the laboratory to minimize downtime.

8.2.2 Any item of the equipment which has been subjected to overloading or mishandling, which gives suspected results, which has been shown to be defective or which fails calibration/verification, shall be clearly identified and taken out of service until it has been repaired. The person making the decision shall provide initials, date and time on the equipment tag and in the logbook.

8.2.3 New equipment shall not be placed into service until properly validated by the technical specialist in the equipment's logbook. It will be clearly identified as not to be used.

8.3 Calibration Equipment

8.3.1 Calibration refers to checking and adjusting variables which can influence the instrument so that the instrument can be used to produce valid information. Calibration does not refer to the standardization or the interim verification of the instrument for a procedure.

8.3.2 Current calibration status (label indicating calibration date and expiration/renewal date) will be placed upon all equipment used for ISO or regulatory testing.

8.3.3 Calibration records must include the signature of the person performing the calibration and a statement attesting to the accuracy/precision of the results. This record is either stored with the administrative personnel or in the form of a lab book with the equipment. Calibration certificates are filed with the administrative personnel.

8.3.4 If a calibration results in a correction factor, all applicable equipment and documents must be updated immediately. The laboratory shall maintain records of these correction factors (in the equipment or calibration log), maintain all original measurements and file a nonconformance, preventative action or corrective action form.

8.4 Standardization

8.4.1 Chemical analysis instruments rely on comparing known chemical standards (both quantitative and qualitative) against unknowns. While the response of these instruments is physical (such as light, current and voltage measurements), they are not calibrated as stated above in 8.3. Chemical standards are either made from pure chemicals or purchased as solutions (refer to the sop for purchases). These standards are then analyzed at several known concentrations and the instrument is standarized to the response that these chemicals produce (refer to section 10.2).

8.4.2 Standardization curves (a.k.a. calibration curves) are prepared by plotting the instrument response versus concentration of the analyte. Curves are generally more applicable to chromatographs or spectrophotometers and are prepared as specified in the methods.

8.4.3 Standard calibration curves, continuing calibration verifications and associated quality assurance and quality control information (QA/QC) are included in the raw data. This data is filed with the administrative staff and electronic data is retained by the lab.

8.5 Support Equipment

8.5.1 All support equipment (devices that are not the test instruments, but are necessary to support laboratory operations) shall be maintained in proper working order. The records of all activities, including service calls, shall be kept in the maintenance log, the calibration log or the designated file. Appropriate requirements following Traceability of Calibration shall be maintained.

8.5.2 BOD incubators, ovens, laboratory refrigerators and freezers, the water stills and Millipore systems are checked daily by assigned laboratory personnel. The information is recorded in a logbook and, if anything is operating out of specifications, the item is tagged and taken out of service. The technical specialist responsible for the area is contacted immediately so that corrective actions can be made. Refer to AQ O-DLYCHK.

8.5.3 All analytical balances are verified daily. Balances that are found to be operating improperly will be tagged and taken out of service. The technical specialist responsible for the area will be contacted immediately so corrective actions can be made. Each balance is thoroughly cleaned and checked at least annually by a balance specialist. Refer to AQ O-DLYCHK.

8.5.4 Mechanical volumetric dispensing devices (except glass micro-liter syringes, Class A burettes and devices not used to generate lab results) are checked for accuracy, using AQ O-PIPCAL, before being put into service to assure that the

device meets the manufacturer's specifications. If exact delivery is required, use AQ O-PIPCAL.

8.5.5 Class A glassware and class A non-glassware is considered to meet the specifications for that type of glassware. Volumetric glassware and glass micro-liter syringes used in the analytical labs shall be Class A.

8.5.6 Non-class A glassware may be used if the manufacturing specifications meet the requirements of the method. If this information is not available, the glassware must be calibrated by the laboratory to demonstrate acceptability. (This also applies to "lab-ware" made of materials other than glass, such as plastic.)

8.6 Traceability and Verification of Equipment

8.6.1 Calibration certificates will indicate, wherever applicable, the traceability of measurements (in SI units) to National Institute of Standards and Technology (NIST) and will provide the measurement results and associated uncertainty of measurements and/or statements of compliance with an identified metrological specification (STP).

8.6.2 Equipment calibration, verification and validation of measurements made by the laboratory, will be traceable to national standards of measurements (in SI), where available. The results of such calibration will be within the specifications required by the application for which the equipment is used.

8.6.3 If traceability to national standards of measurement is not applicable, WATS will provide evidence of correlation of results by participation in a suitable program of inter-laboratory comparisons or proficiency testing whenever possible and practical.

8.6.4 For chemical analysis, chemicals and chemical solutions that are of a different lot number or different manufacturer (a second source) are compared to the calibration standard to verify calibration, when available. If possible and practical, solutions traceable to national standards (e.g. NIST) will be used. NIST also provides standard reference materials.

8.6.5 Reference standards of measurement held by the laboratory (such as Class 1 or equivalent weights, traceable thermometers, wavelength standards, etc.) are used for calibration only. Reference standards of measurements shall provide, whenever possible, traceability to a nation standard of measurement, preferably NIST.

Chemical Standards and Reagents

9.1 The quality of chemical standards and reagents must be appropriate for their intended use with additional considerations to quantity, where purchased, how received and how stored. The grade of the reagent used (including water) is stated in the method, together with guidance on any particle precautions and hazards that should be observed in its preparation, storage and use.

9.2 Documentation and labeling are vital to insure the accuracy and validity of the test data, providing traceability and stability information. The laboratory shall retain records for all chemicals, which include manufacturing/vendor identification; lot number; manufacturer's certificate of analysis and/or purity (certificates of analysis must be retained for standards); date of receipt; recommended storage conditions; date opened and by whom; and an expiration date. In addition, original reagent containers shall be labeled with the date received and opened, by whom and an expiration date. In some situations, the Certificate of Analysis is the label on the reagent bottle.

9.3 Where the quality of a reagent or analytical standard is critical to the test, the new batch is verified against the outgoing batch before use, provided that the outgoing batch is known to still be serviceable. For reagent and chemical standard preparation, detailed records (laboratory logbooks and service request data sheets) shall be maintained, including traceability to purchased stock; method of preparation; date of preparation, replacement and expiration; and preparer's initials. The expiration dates of chemicals will be monitored per the manufacturer's recommendation. All reagents made from these chemicals will also retain the same expiration date as the chemical. If there is no expiration date given, the best judgment of the analyst will used.

9.4 The storage and eventual disposal of chemicals and reagents are documented in the "A&T Chemical Hygiene Plan" and comply with Weyerhaeuser's Chemical Management Department.

References
Chemical Hygiene Plan
Weyerhaeuser's Chemical Management Dept. http://www.weyer.com/chemmgmt/

Quality Control Testing Procedures

10.0 Quality Assurance (QA) in sample testing and analysis relies on following quality control (QC) procedures on a systematic basis. Besides the test method requirement of standard calibration, additional testing of method blanks, control samples, duplicates, lab control spikes and matrix spikes provide QA/QC information.

10.1 Samples are prepared in a batch.

- The number of samples per batch is defined in the method. This preparation batch is composed of samples of the same matrix (i.e., soil, water, pulp, etc).
- These samples are prepared together with the same process, personnel, and lot(s) number of reagents.
- These samples are prepared with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours, unless specified otherwise in the test method.
- While the batch size can vary, the QC requirements are that a method blank and/or instrument blank and lab control spike (or reference sample) are prepared with the samples whenever possible.
- At least 1 duplicate or matrix spike are required for every 20 samples (the total samples can be from multiple batches), unless specified differently in the method.
- An analytical batch is composed of prepared or non-prepared samples (extracts, digestives or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
- If the MS, MSD or duplicate are diluted, the sample must be also diluted.
- An effort will be made to spike non regulatory samples.

10.2 Standards

10.2.1 Standardization of the instrument is done by analyzing different known amounts of a stock reagent standard (which contains the analyte of interest). This generates response factors (a value that is produced by comparing the response of the instrument versus the amount of the analyte). These response factors then can be averaged or subjected to a curve-fitting calculation. This mathematical summarization of the response factors generates a formula, which is then used to calculate a concentration for samples.

10.2.2 To cross check the fit of this calculation, the standards are subjected to the calculation and a percent difference is determined from the theoretical concentration versus the true concentration. A good average would have a standard deviation of less than 15% and a good cross check of the lowest

calibration concentration would be less than 10%. (Of course, this required QC criteria would be specified in the method.)

10.2.3 Different calibration levels are required to generate a valid average response factor. More levels are required to generate calibration curves. It is recommended that 5 for average, 5 for linear, and 7 for any quadratic are used. In general, the regression correlation coefficient, r, (how well the data fits the curve), should be at least 0.990. The coefficient of determination, r2, should be at least 0.980. Again, these QC criteria would be specified in the method. (Some labs run a "0" concentration as the fifth calibration point.) Do not include the 0 concentration as part of the curve, unless specified in the method.

10.2.4 A further calibration criterion would be the upper and lower range of the concentrations. The low concentration represents the quantitation limit of the method, and is specified in the method. The upper concentration represents the maximum amount of analyte that can generate a valid number. (The Metals Laboratory runs a linearity check annually for the upper acceptable concentration, which is magnitudes above the highest calibration concentration.) Any sample that generates a response greater than this amount needs to be diluted and re-analyzed. If PQL is used to report the quantitation limit, include the PQL as part of the calibration curve.

10.3 Detection Limits

10.3.1 The reported lower detection limit may be the instrument detection limit, method detection limit (MDL), practical quantitation limit (PQL) or the low standard quantitation limit. The method (or client) will determine which limit to report.

10.4 Calibration and Verification

10.4.1 After calibration, a continuing verification standard (usually the mid-concentration) is analyzed at some frequency (in general, once every 10 samples) to indicate that the calibration is still valid. Again, a good percent difference would be 10% from the expected value. (This required QC criteria would be specified in the method.)

10.4.2 Some methods only require valid calibration verification before analyzing samples, while other methods require valid calibration verifications before and after sample analysis.

10.4.3 An initial calibration verification (ICV) is independent from the calibration standards and validates the calibration curve. This is analyzed once with a new calibration curve. (Chromatography substitutes the ICV with the LCS.)

10.5 Blanks

10.5.1 Preparation of a method blank is required (as if it were a blind sample) and is analyzed with all samples.

10.5.2 This blank establishes that no contamination occurred during the sample preparation and analysis. The blank also establishes that there is no contamination from the reagents. In general, if an analyte is detected in the blank at a concentration less than ½ of the quantitation limit, the analyte is not reported (The Metals Lab doesn't report the analyte if is less than the quantitation limit.) The method blank also can be used to determine realistic detection limits by indicating the background level of the process.

10.5.2 Instrument blanks are also analyzed at some frequency (either before or after the verification) to establish that there is no cross contamination between analyses. Additional instrument blanks are run after samples that are suspected of causing contamination. Instrument blanks can also be used to help clean the system.

10.5.3 Field and trip blanks serve to indicate that no contamination occurred during the sampling and shipping of samples. These types of blanks are dependent on the method sampling requirements.

10.6 Control and Reference Material

10.6.1 Lab control spikes, initial calibration verifications and reference materials are used to monitor the overall precision and bias of a method.

10.6.2 A lab control spiked sample (LCS) is a blank matrix spiked with analyte, indicating if the preparation of the samples is valid.

10.6.3 Reference materials are used to verify that the method gives valid results for a given matrix. These reference materials are samples that have been analyzed by multiple laboratories (or if unique to Weyerhaeuser, analyzed multiple times by WATS) and have established acceptable ranges for the analyte of interest. Analyses of reference materials provide an external (referred) source of precision and bias of a method on that type of material.

10.7 Spikes Required

10.7.1 A spike is a standard reference material added to a sample and carried through the procedure in order to see if adequate recovery can be obtained. This helps to evaluate the performance of a procedure being developed or the effect of a new sample matrix on analyte recovery. Poor spike recovery indicates that the procedure may not be effective in analyzing the sample.

10.7.2 There are 4 types of spikes:

- Matrix spike (method spike) – this spike consists of target analytes that are carried through the entire procedure.
- Surrogate spike – this spike is an analyte that is similar in chemical characteristics to the target analytes, but is not the target analyte. It is added to all of the samples, method blank and QC samples. Poor surrogate recovery in the method blank and the lab control spike indicate a problem with the sample preparation procedure.
- Analysis spike – this spike is added to the prepared sample, usually after digestion or extraction, at the instrument level (as opposed to being carried through the overall method). It is used to check for interference problems in a specific matrix.
- Field spike – this spike is a matrix spike that is added to the sample in the field. It is used to evaluate the overall accuracy during sampling, transporting and preservation.

10.8 Replicates Required

10.8.1 Replicate (duplicate) samples generate important QC information of sample matrix precision. The method may allow matrix spike duplicates in place of sample duplicates.

Quality Control Monitoring by Control Charts

11.1 For effective Quality Control/Quality Assurance, there must be a means of monitoring and examining quality control (QC) data. Examining QC data on the batch level can give information of whether the batch was valid (or indicate a problem with the batch), but does not give a true idea of the process validity.

11.2 QC control charts are a visual way of monitoring the process. It gives information on precision of data, and it can show systematic departures of the data from the mean (i.e. incremental changes that reflect a pattern to this change).

11.3 Information gained from interpreting control charts provides diagnostic information and indicates the process capability, offering insight into improvement of productivity (by being able to see problems occurring before having a significant impact on the data) and improvement of the quality of the data (by knowing the true variability of the data).

11.4 WATS' laboratories have a broad range of capabilities and many of our samples are unusual or even unique. Tests that are not routinely run (i.e. run occasionally, such as monthly, quarterly or annually) are expected to follow WATS' quality control policy and procedures, where the emphasis will be placed upon spikes and replicates being run. However, with the possible exception of the standards, traditional control charts are not expected to be generated.

11.5 Procedure

11.5.1 Control charts follow the SOP for control charting. The data points are plotted in the order that they are generated (sample value versus time or sample number). The mean, warning limits (2 standard deviations from the mean) and control limits (3 standard deviations from the mean) are calculated (from the first 20 data points) and plotted on a standard control chart.

Detection and Quantitation Limits

12.1 Test methods used should provide a detection limit that is appropriate and relevant for the intended use of the data. A new detection limit must be determined each time there is a significant change in the test method or instrument type. If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

12.2 Since the laboratory normally has no control over the samples before they are received, limits for the complete testing protocol are usually not addressed. However, interest in this is highly encouraged. Field blanks and trip blanks can sometimes point out highly significant factors that can affect the true limits of a testing program.

12.4 Outlier Determination with Small Data Sets

12.4.1 The process for outlier determination is outlined in the SOP **Outlier Determination with Small Data Sets**- AQ O-OUTLIER that describes using Dixon's "test" procedure and the "T" table to evaluate if a single value is an outlier. Before using the SOP for outlier determination to evaluate whether a test result is an outlier, determine whether there are any non-statistical reasons that the result should not be included in a data set. These can include sample defects, samples not being 'representative', failure of a sample to respond properly to a test, and failures in the testing process.

Rounding

13.1 Significant figures will be within the constraints allowed by computer programs. Some programs operate within computer systems over which the laboratory has no control.

13.2 Data need to have the proper number of significant figures. This is generally the number of digits necessary to express results consistent with the measured precision and accuracy, customarily, the digits that are 'known' plus the first uncertain digit. In some cases, the number of significant digits may be limited by the ability to measure. Where possible, the number of significant figures are written into the method, or a means of deriving the number should be described in the method or in the SOP.

13.3 All digits from an analytical measurement should be recorded, unless an instrument manufacturer says to truncate them at a certain point. They are all original data and need to be saved. Rounding off should not be done until the data are analyzed. (Some reason may need to be applied here. Some instruments print out a large number of digits, of which only 1 or 2 may be significant. In such cases, it would be impractical to carry all of the digits.)

13.4 All numbers that start with a decimal point should begin with a zero to the left of the decimal. An exception to this is in systems where computers automatically leave the digit out.

13.5 All zeroes after the decimal point and to the left of the number are not considered significant. Zeroes carried to the right of a number following a decimal point are counted as significant. Zeroes to the right of a number preceding a decimal are generally considered unknown; thereby they are not usually significant.

300	Unknown number of significant figures, may be 1,2, or 3.
300	3 significant figures
0.0028	2 significant figures
$5x10^4$	1 significant figure
0.30020	5 significant figures

13.6 An alternate to using the exponential form is to change the units for reporting. 5400 μg/L could be reported as 5.4 mg/L instead of 5.4×10^3 μg/L. Whenever there is a wide range of values for a given unit in a report, it is advisable to have a comment stating the number of significant figures.

13.7 When making product or quotient calculations, the results can have no more significant figures than the value in the equation with the fewest significant numbers. Some equations have factors in which not all the significant digits may be displayed. In some cases, an absolute factor, such as 100 %, is used in which

the number of significant figures can be considered infinite. If there is any doubt about what should be done in some cases, discuss it with the Quality Assurance/ Quality Control Focused Team Members.
Example:

$$\% = (((38.53g)/mole)(0.1031N)(0.045L))X100)/3.0893g = 0.057864\%$$

Here the 0.045 L is the limiting factor with two significant figures, so 0.057864 would become 0.058.

13.8 Whenever the absolute magnitude of a number is greater than that of the limiting number, the significant digits may be increased by one. In this case, the last digit is written with a bar below it to indicate the last digit is in doubt.
For example:

$$((40.08)(751))/((142.3)(0.45572)) = 4328$$

13.9 When making a calculation involving addition or subtraction, the location of the decimal point determines the level of significance. The sum or difference can have no significant figure farther to the right than the number whose last significant digit is farthest to the left.
For example:

4 significant figures	3 significant figures
104.3	40 100
21.72	223
2.349	1.03
2.672	0.237
0.1276	10.1
10.22	3.47
141.3886	40337.837

These then become 141.4 and 40 300 when rounded off.

13.10 When making calculations involving logarithmic values (i.e., pH calculations), the number of digits in the mantissa of log *x* is equal to the number of significant figures in *x*.
For example:

log 339 = 2.530

Where 2 is the character and 530 is the mantissa.

When converting a logarithm to its antilogarithm, the number of significant figures in the antilogarithm should equal the number of significant digits in the mantissa.
For example:

Antilog (-3.42) = $10^{-3.42}$ = 3.8 x 10^{-4}

Where -3 is the character and 42 is the mantissa

13.11 If numbers are to be used as a factor in a calculation, an extra significant figure may be carried. Thus, 0.058% would become 0.0579% and 141.4 would become 141.39.

13.12 Except for detection limits, the following rules will be used in rounding to the last significant digit in a number.

1. When the digit after the last significant digit is less than 5, all the digits after the last significant digit will be dropped and the retained digit will remain unchanged.
   For example, 3.264 would round to 3.26.

2. When the digit after the last significant digit is greater than 5, all the digits after the last significant digit will be dropped and the retained digit will be increased by one.
   For example, 3.267 would round to 3.27.

3. When the digit after the last significant digit is exactly 5, the 5 will be dropped and the last significant digit will increase by one if its preceding digit is odd. The preceding digit will remain the same if it is even.
   For example, 3.25 would round to 3.2 and 3.35 would round to 3.4.
   Numbers farther to the right generally have no significance.

13.13 Sample detection limits will be reported to only one significant figure. When rounding digits for detection limits, round up to the nearest significant digit. MDL (Method Detection Limit) determined by 40CFR136 (Federal Clean Water Act) may be stated to 2 figures since it is a statistic, not an actual detection limit.

13.14 A statistic of a data set (such as the mean and standard deviation of several analyses) may have one more significant digit than the samples used to calculate it since it is a statistic and not an analysis. However, this laboratory does not view the mean of duplicates, and in many cases, triplicates, as statistical.

13.15 Calculated confidence limits will only be stated to the same level of significance that the data will have. For example, 17.5 can have a confidence interval of ±0.2, not ±0.23 or ±0.232. The standard deviation, a statistic, can have one more

significant figure than the confidence interval. Thus, if 0.23 were a standard deviation, it would be acceptable. It is not acceptable as a confidence limit.

Data Recording, Control, and Verification

14.1 The record keeping system must allow historical reconstruction of all laboratory activities that produced the resultant sample testing data.

14.2 Testing results for all samples, standards, and quality control (QC) are based on actual results as performed.

- Documented data must match actual data. Making up data, substituting, altering or correcting data or other information - creating data for tests that were not performed or creating information that is not factual - is not permitted.

- Entries in records shall not be obliterated by methods such as erasures, overwritten files, or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. All original data electronically entered shall not be altered except through a process that leaves an electronic audit trail back to the original information. For some equipment, this may not be possible. In such cases, the original and the corrected version shall be retained. The original may be in the form of a print out.

- Copying and destroying valid original data is not permitted. If copying must be done to make things more clear, legible, or understandable, the original must still be retained.

- Print outs of data used to store or represent electronic data/information must reflect the actual conditions under which the data were generated.

14.3 All original documents and data shall be signed, or initialed, by responsible staff and retained for the records. This includes, but is not restricted to, all original raw data, whether hard copy or electronic, calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records). This does not apply to information that is not original, that can be reconstructed from the original, and that are otherwise not acted upon.

14.4 Multiple pages of continuous electronic print-outs, such as GC/MS, FQA, ICP/MS, ion chromatography, only need to be signed at the beginning of a series. Hand-altered material within those pages should be acknowledged.

14.5 A series of pages of hand-written similar material should be initialed by the generator. Witnessing and dating may be done only on the first page of the series.

14.6 The recorded date and time of collection, preparation, or testing must match the actual date and time that the action was performed. These records shall include the identity of personnel involved in (sub) sampling, preparation, calibration, testing, and checking of results. This identification is normally done by initials, signature, or electronic identification in the LIMS.

14.7 The reason for the signature or initials should be clearly indicated in the records such as “sampled by” (SB), “prepared by” (PB), “analyzed by” (AB), “tested by” (TB), or “reviewed by” (RB).

14.8 All original data shall be recorded directly, promptly, and legibly in permanent, indelible ink, except those that are generated by automated data collection systems. Such information shall be identifiable to a specific service request. Blue ink color is preferred in order to differentiate original data from copier data.

14.9 Calibration points can only be rejected for inclusion in the calibration curve if a known error was made or if a statistical evaluation indicates that a point can be discarded. Points may be discarded at the upper end of the curve if the linear range of the detector has been exceeded. For these cases, the laboratory must dilute samples that exceed the highest point of the calibration curve. Points can be discarded at the lower end of the curve if the detector is not producing a response. For these cases, the laboratory’s reporting limit must be adjusted accordingly.

14.10 Instrument peaks, slopes, or changes in slope, must be consistently measured and reported according to proper techniques. For chromatography, this generally is baseline to baseline, valley to valley or a combination of the two.

14.11 Any knowledge of testing or sample problems must be documented and communicated to laboratory management and the client (if appropriate).

14.12 Any knowledge of unethical or illegal behavior or actions must be fully communicated to laboratory or corporate management. Concealing a known unethical or illegal behavior or action is considered complicity.

14.13 All calibration/QC data associated with sample tests must be documented.

14.14 Computer manipulation is allowed only for warranted reasons and any manipulation should be minimal and traceable. Removal of computer operational codes is not permitted. Performing multiple (more than two) calibrations or complete initial calibration runs (including calibration verifications, LCSs, spikes, duplicates and blanks) until one barely meets criteria is not allowed.

Laboratory Reports

15.1 The results of each test or series of tests carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively, and in accordance with any specific instructions in the test procedure.

15.2 Format and Contents

15.2.1 Laboratory reports will be written in accordance with OP REPORT GEN.

15.3 Opinions and Interpretations

15.3.1 Opinions, interpretations, or amplifications, not based upon a process described in a procedure, shall be clearly identified. Laboratory should document the basis upon which opinions, interpretations, or amplifications have been made.

15.4 Reports with Errors

15.4.1 The laboratory shall notify clients promptly of any event, such as the identification of defective measuring or test equipment, nonconformance, or other event casting doubt on the validity of results given in any test report or report amendment. Any regulatory work must be recalled. Work which is not ISO or regulatory in nature is subject to laboratory/client agreement on a course of action.

15.4.2 A corrected report shall be created and be labeled as a 'Revised Report.' A copy of all revised reports shall be kept with the original data (SR).

15.5 Electronic Transmission

15.5.1 The laboratory shall ensure that, where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, staff will follow procedures that ensure that confidentiality is preserved.

15.5.2 All official reports, such as those for regulatory use, sent electronically shall be sent in a form that cannot be altered, such as a print-only PDF file. There will be copies of original reports retained with the data.

15.5.3 Electronic data files containing user-friendly data may be sent to the clients requesting them. However, an official report will also be sent.

15.6 Confidentiality

15.6.1 INTERNAL. Data generated by the laboratory is generally considered to be confidential to a given client or client set. It will not be shared with others

without the permission of those submitting the work (or permission of one who is an agent of the submitter). A client set is taken to be a group or association of individuals working on a common project or purpose, e.g. associated members of an environmental department at a given mill. It generally does not mean a production person from one mill being analogous with a similar individual at another mill. Sometimes proprietary concerns on the part of the Corporation's customers may preclude any exchange of information within the Corporation's business units.

15.6.2 EXTERNAL. Work from customers external to Weyerhaeuser, and not associated with Weyerhaeuser (such as by subcontracting), is considered confidential and not to be shared with anyone other than those specific customers without their written permission. However, since we are an internal corporate laboratory, we will not take exceptional measures, beyond those normally used to process data and to safeguard information. External clients should be made aware of this in writing.

15.6.3 ATTORNEY-CLIENT PRIVILEGED. In some instances, data may be attorney-client privileged. This may involve revealing information only to a specified attorney. This can include all raw data, any reports, unused sample, etc., which may be required to be given to those attorneys. In such instances, data and information shall not be revealed to any persons, including Weyerhaeuser employees, except as necessary to execute the work.

Acronyms, Glossary, & Qualifiers
These terms are presented here as a reference and may be found in the quality manual, laboratory policy and guidelines, or they may be used by customers.

	Acronyms
AA	Atomic Absorption
AM	Analytical Method
ABM	Absorbent Products Methods
AR	Analytical Reagent Grade Chemical
ASTM	American Society for Testing and Materials
ASR	Archived Service Requests
CAA	Clean Air Act
CAL	Calibration standard (EPA)
CC	Correlation Coefficient. (See r.)
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CD	Cross Direction
CL	Control Limit(s)
CLP	Contract Laboratory Program (also used to refer to projects needing CLP data packages)
COC	Chain of Custody
COD	Coeffficient of Determinination (See r^2.)
COV	Coefficient of Variation
CQLV	The low level Continuing Calibration Verification (CCV)
CRDL	Contract Required Detection Limit
CRM	Certified Reference Material
CVS	Calibration Verification Standard
CWA	Clean Water Act
°C	Degrees Celsius (a.k.a. centigrade)
df	Degrees of freedom
DL	Detection Limit
DMR	Discharge Monitoring Report (Report required by permitted discharger.)
DOT	Department of Transportation
DQO	Data Quality Objectives
FDA	Food and Drug Administration
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
GC	Gas Chromatography
GC/MS	GC Mass Spectrometer
GFAA	Graphite Furnace AA
HAP	Hazardous Air Pollutant (CAA)
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification

ICP or ICAP	Inductively Coupled (Argon) Plasma
ICPMS	ICP Mass Spectrometer
IDC	Initial Demonstration of Capability used to assure that lab can analyze unknown samples via a new, unfamiliar method prior to obtaining experience with the method.
IDL	Instrument Detection Limit
IDP	Initial Demonstration of Proficiency See Initial Demonstration of Capability.
IPAR	Initial Demonstration of Method Performance (See IDP.)
IPC	Institute of Paper Chemistry
IR	Infrared Spectrometry
ISO	International Organization for Standardization
k_x	See Glossary.
K	Kelvin
LCS	Control or Laboratory Control Sample
LD	Analytical Detection Limit
LOD	Limit of Detection (or Detection Limit)
M^+	Molecular ion (used in mass spectrometry)
MD	Machine direction
MDL	Method Detection Limit
ML	Minimum Level
MML	Minimum Measurement Level
MRL	Minimum Reporting Level
MS	Matrix Spike
MOE	Modulus of Elasticity
MOR	Modulus of Rupture
MSD	Matrix Spike Duplicate
MS/MSD	Matrix Spike/Matrix Spike Duplicate combination
m/z	mass-to-charge ratio (used in mass spectrometry)
N	Normal (used in chemistry) (N is preferred form.)
NBS	Former acronym for NIST. The National Bureau of Standards.
NELAC	(See NELAC in glossary.)
NELAP	(See NELAP in glossary.)
NIST	National Institute for Standards and Technology
NPDES	National Pollutant Discharge Elimination System. (Regulations under the CWA.)
NV	Non-volatile
ORB	Original Record Book
ORF	Original Record File(s)
OPR	Ongoing Precision and Recovery standard (See Laboratory Control Sample.)
PAPRICAN	Pulp and Paper Research Institute of Canada
PB	Preparation Blank
P & B	Precision and Bias
PBMS	Performance Based Measurement System
PT	Proficiency Test Sample
PQL	Practical Quantitation Limit

QA	Quality Assurance
QC	Quality Control
QL	Quantitation Limit
QA/QC	Quality Assurance and Quality Control
r	Correlation Coefficient
r^2	Coefficient of Determination
RCRA	Resource Conservation and Recovery Act. (Solid and hazardous waste regulations)
RM	Reference Material
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
s or sd	Standard Deviation
SOP	Standard Operating Procedure
SM	Standard Methods published by APHA
SRM	Standard Reference Material
TAPPI	Technical Association of Pulp and Paper Industries
TIC	Tentatively Identified Compound
$U = k\ u_c(y)$	Expanded uncertainty
$u(x_i)$	Standard uncertainty
$u_c(y)$	Combined standard uncertainty
VER	Calibration verification standard
WL	Warning Limits

Glossary

Acceptance Criteria — Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Absolute Magnitude — Used in statistics, magnitude refers to “size” or the strength of a relationship between variables. Term usually references the absolute value.

Accuracy — The degree of agreement of a (analytical or test) result with the true* value. The accuracy of a result is affected by both systematic errors (bias) and random errors (precision). Accuracy is a data quality indicator and can result from any operation from sampling through to the final number. It should be noted that some writers use "accuracy" to denote only systematic error (bias).

*’true’ value may be an accepted value; e.g., the value from a standard supplied by NIST. An example is a pH buffer.

Action Limit — See Control Limit.

Analyte — Denotes "that which is to be analyzed for”. Only covers chemical analyses, and not physical or biological determinations.

Analytical Error	The error, E, of an analytical or test result, R, is defined as E =R-T, where T is the true value.
Analytical Method	See Method.
Analytical Detection Limit (LD)	See Detection Limit.
(Analytical) Reagent (AR) Grade	See Reagent (AR) Grade.
Analytical Response	A numerical observation which is obtained when a portion of a sample is presented to a measurement sub-system, e.g., spectrophotometric measurement of the absorbance of a solution. The magnitude is related to the amount or concentration of the determinant (analyte) in that portion.
Analytical Result	See Test Result.
Analytical Support Equipment	See Support Equipment.
Analytical System	Denotes a combination of analyst, analytical method, equipment, reagents, standards, laboratory facilities, and any other components involved in carrying out an analytical procedure.
ASR	Archived Service Request, formerly known as ORB (original record book).
Batch	This usually refers to a set of consecutive determinations (analyses) made without interruption. The results are usually calculated from the same calibration curve or factor. Also called a run. These are usually samples prepared and/or analyzed together with the same process and personnel, and using the same lot(s) of reagents. Generally more appropriate for the analytical lab. Analytical batch is usually composed of prepared samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Preparation batch is composed of one to 20 (environmental) samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours.

Blank — A sample, normally reagent water (but may be another reference matrix) not containing added analyte, intended to estimate the analytical responses attributable to factors other than the parameter of interest. Generally, it is a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. Blanks are analyzed at the same time as and in an identical manner to samples.
An example would be pure water analyzed to determine the blank for water matrices.
Field Blank – A blank exposed to the same field condition as a sample. Primarily to determine if samples collected in the field are being contaminated by material present in the sampling and transportation environment, or preservation process. Examples include atmospheric contamination during bottle filling or contamination from field filtering or pumping.
Instrument Blank – A blank (e.g., distilled water) processed through the instrumental steps of the measurement process to determine instrument contamination or instrument responses not attributable to a given parameter.
Method Blank – A blank processed through all steps of the analytical (or test) processes to determine any contribution of the reagents and any of the involved analytical (or test) steps, including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples.
Reagent Blank – Generally equivalent to the method blank.
Trip Blank - A blank sample that accompanies bottles being shipped and being returned with sample. It purpose is to determine any contribution to the sample by the activities associated with sampling and transportation. (Usually this is only for the VOC samples.)

See also Negative Control.

Blank Spike — See Laboratory Control Sample (LCS.) A term that is used in GC, but is not a recommended usage.
Also, it has been known to be used for the 'spike' containing no analyte, or 'zero,' when performing a 'method of standards addition'.

Blind Sample — A sub-sample for analysis with a composition known to the submitter. The analyst or laboratory may know the identity of the sample but not its parameter composition. Generally, it is used to test proficiency in the execution of the measurement process. (See also Double Blind.)

Calibrate	To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected measurements.
Calibration	The set of operations establishing, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system, or values represented by a material measure, and the corresponding known values of the parameter being measured. (Example: Micrometer gives the same value as NIST traceable caliper block.) NOTE: Commonly and improperly confused with standardization.
Calibration Curve	The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. May be linear or non-linear. Generally, it is more appropriately called a 'standard curve'.
Calibration Standard(s)	Solution(s) of known concentration which are used in the calibration (standardization) procedure to determine the relationship between response and concentration. These are generally solution prepared from the stock standard solutions, as well as internal standards and surrogate analytes. Also, materials of known value used in calibration, such as weights or thermometers.
Calibration Method	Defined technical procedure for performing a calibration.
Calibration Verification Standard (CVS)	A calibration standard that is run to verify the integrity of an initial calibration curve which is not run on the day of analysis. (EPA) The mid-point calibration standard (VER) that is used to verify calibration
Celsius	Official term for centigrade. Equal to 9/5(°F) + 32.
Centigrade	Common term for Celsius (°C).
Certified Reference Material (CRM)	A reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.
Chain of Custody (COC)	An unbroken trail of accountability that documents the physical security of samples, data, and records.
Coefficient of Determination	Equal to square of the correlation coefficient, or r^2. Should not be confused with correlation coefficient, r. Coefficient of determination is the <u>proportion</u> of the variation in the dependent variable (y) that can be explained by relating y to x, the independent variable. In an analytical laboratory setting, y is usually the instrument response (area, counts, absorbance, etc.) and x is usually the standard

	concentration. (Proportion of explained variation/ total variation.) CAUTION: This is a measure or indication of the degree of linear (explained) relationship. It is not necessarily the best or most appropriate fit for the data. For example, the line may actually be a curve rather than straight. If so, the conclusion would be erroneous.
Coefficient of Correlation	See Correlation Coefficient.
Coefficient of Variation (COV)	See Relative Standard Deviation.
Combined standard uncertainty (ISO)	A value approximately equal to the square root of sum of the variances from the standard uncertainties. In other words, this is the pooled standard deviation of standard uncertainties.
Confirmation	Verification of the presence of a component or property using an analytical or testing technique that differs from the original method. Examples include: second column confirmation, use of an alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, and additional cleanup procedures.
Continuing Calibration Blank (CCB)	An ICB that is run after every CCV.
Continuing Calibration Verification (CCV)	An ICV that is run periodically throughout an analysis. For CLP it is done after 10 analyses and at the end of a run.
Continuing Calibration, Low Level (CQLV)	The low level Continuing Calibration Verification (CCV).
Contract Required Detection Limit (CRDL)	Low level standard at or near the IDL, depending upon CLP contractual requirements for the specific analytes.
Control (Quality Control Sample)	A material of known or established value, independent of the standard, which is used to check the precision of analyses, and bias attributable to calibration. It is carried through the some or all method processes in the same manner as a sample. The matrix should match that of the sample as closely as possible. Independence can be established by using a different vendor or lot number.

It is assumed to be carried through the entire analytical or testing process unless it accompanied by a modifier; e.g.; instrument control.

Control Limit(s) (CL)

Limit of control that is specified by a value on a control chart. If the value is exceeded, then corrective action must be taken. Control limits are usually placed at ±3σ distance from the expected value. A better term would the 'normal' limit since control charts reflect what is normal for a procedure as it has been performed.

Corrective Action

Action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

Correlation Coefficient

It is equal to r in the below formula. It should not be confused with r^2, which is called the coefficient of determination. Correlation coefficient underestimates the degree of linearity.

In mathematics, and in particular statistics, the Pearson product-moment correlation coefficient (r) is a measure of how well a linear equation describes the relation between two variables X and Y measured on the same object or organism. It is defined as the sum of the products of the standard scores of the two measures divided by the degrees of freedom.

The formula for Pearson's correlation takes on many forms. A commonly used formula is shown. The formula looks a bit complicated, but taken step by step, it

$$r = \frac{\Sigma XY - \frac{\Sigma X \Sigma Y}{N}}{\sqrt{\left(\Sigma X^2 - \frac{(\Sigma X)^2}{N}\right)\left(\Sigma Y^2 - \frac{(\Sigma Y)^2}{N}\right)}}$$

is really quite simple.

A simpler looking formula can be used if the numbers are converted into z scores:

$$r = \frac{\Sigma z_x z_y}{N}$$

where z_x is the variable X converted into z scores and z_y is the variable Y converted into z scores.

Criterion of Detection

The smallest concentration which can be distinguished from a blank with no more than a specified chance of reporting a false positive.

Cross direction (CD)

See Machine Direction.

Data	See Raw Data.
Data Quality Objectives (DQO)	Qualitative and quantitative statements of the quality of data needed to support specific decisions or regulatory actions.
Data Reduction	The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useful form.
Detection Limit (DL)	The smallest amount of a parameter that can be distinguished in a sample by a given measurement procedure throughout a given (e.g.; 0.95) confidence interval. OR The lowest result of a concentration measurement that is not consistent with the hypothesis of zero concentration at a specified level of confidence. The American Chemical Society states it is the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence. (NELAC) See also Limit of Detection.
Degrees of Freedom (df)	A whole number expressing the amount of information available to a particular estimate; it is generally the number of independent results less the number of constraints; that is, what else has to be estimated from the same set of results.
Double Blind Sample	A blind sample submitted to evaluate performance in which also the identity is unknown t o the analyst. (See Blind Sample.)
Duplicate (Analyses or Test)	A replicate restricted to two sub-samples. See Laboratory Duplicate.
Error	1. Deviation of an observed or measured value from the expected or true value. 2. A (gross) mistake or blunder.
Expanded uncertainty (ISO)	Quantity defining an expected interval about a result of a measurement that encompasses a large fraction of the probability distribution characterized by the measurement result.
External Standard Calibration	The use of independently purchased and prepared standards to determine the relationship between response and concentration. Also called instrument calibration verification or ICV.
Felt side	In making paper, when the sheet is picked up from the screen (called a 'wire'), it is carried through the drying section of the paper machine on a fabric called a 'felt.' The felt side is opposite the wire side. (See wire side.)

Holding Times (Maximum Allowable)	The maximum times that samples may be held prior to analysis and be considered (legally) valid. For the CWA, this is stated in 40CFR136. For non-regulatory samples, other procedures may be used, such as in ASTM D4515. (NOTE: For many of the parameters, the legal holding time is arbitrary and does not reflect actual deterioration.)
Initial Calibration Blank (ICB)	A zero standard that is run just after the ICV.
Initial Calibration Verification (ICV)	See External Standard Calibration. For CLP, it must be at a concentration other than that used for calibration and lie within the range of the calibration standards. CLP requires it to be run right after instrument calibration.
Initial Demonstration of Method Performance	Initial Demonstration of Precision and Accuracy. See Initial Demonstration of (Analytical) Capability.
Initial Demonstration of Precision and Accuracy	See Initial Demonstration of Capability.
Initial Demonstration of (Analytical) Proficiency (IDP)	See Initial Demonstration of Capability.
Initial Demonstration of (Analytical or Testing) Capability	Procedure to establish the ability of the analyst or tester /laboratory to generate acceptable accuracy and precision which is included in many analytical or testing methods. In general, the procedure for EPA methods includes the addition of a specified concentration of each analyte (using a QC check sample) in each of four separate aliquots of laboratory pure water. These are carried through the entire analytical procedure and the percentage recovery and the standard deviation are determined and compared to method specified limits.
Instrument Blank	See Blank.
Instrument Detection Limit (IDL)	1. This is the <u>instrument</u> detection limit, or the LOD for a specific instrument. It can apply to the limit of the ability of an instrument to measure under pure ideal systems. It can also refer to the instruments abilities under a specific type of matrix. For example, an IDL for distilled water with atomic absorption

would be different from one in a highly saline matrix. Care must be taken not to confuse the two. In some instances, the IDL may be the same as the MDL.
2. Under CLP, a value equal to 3 times the average of the standard deviations of an analyte in water at 3 to 5 times the expected IDL, run in 7 consecutive measurements on 3 non-consecutive days. This may differ for other contracts.

Interference	A systematic error (bias) in the analytical or test result caused by the presence of a substance in the sample (or added to the sample during analysis).
Interference Check Standard	A standard solution containing a broad array of elements (metals) that is run periodically to verify the inter-element spectral interference correction routine for plasma emission spectrographs.
Internal Standard	A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical method.
Internal Standardization	A calibration procedure in which the responses of analytes are determined relative to internal standard(s) which is (are) added to every sample. Two solutions - calibration and spiking - are required. Used to reduce bias due to calibration.
International Organization for Standardization	1. An organization comprised of a specialized system for worldwide standardization going by that name. 2. Within the confines of WATS QA manual: a. ISO followed by a standard number refers to that identified standard. b. ISO used as an adjective indicates a need to follow the dictates of that organization, mainly the requirements indicated in ISO 9001.
Isotope Dilution	A type of internal standardization procedure in which the internal standards are isotopically labeled analogues of the analytes. Characterized by very low bias due to calibration.
k_x	A number from Student's t table (for two-tails) that reflects the degree of confidence. For 95 %, for larger data sets, it is equal to 2. 'x' designates type 'A' or 'B' from A6.4.2.1.
°K (Kelvin)	Temperature scale using units of measure equivalent to centigrade degrees, but which begins at –273.16 °C. 20 °C equals 293.16 °K. Used in certain chemical calculations; e.g., Nernst equation used in ion selective electrode work.
Laboratory Control Sample (LCS)	See Control. For CLP, it must be run before any samples. An LCSW is for water and an LCSS is for solids.

Laboratory Duplicate	A duplicate taken from the same container under laboratory conditions.
Limit of Detection (or Detection Limit)(LOD)	This is the lowest level that a measurement can be determined to be statistically different from a blank. Generally, this is taken as the signal that is equal to the blank value plus three times the standard deviation of the blank. However, this is an arbitrary convention, and it may need to be replaced, depending on the method or situation. In some instances, greater confidences may be needed, calling for four, or even five, standard deviations. In other cases, a "blank" signal may not be produced, making another approach necessary. Every effort will be made to follow recognized conventions for the type of analysis. The smallest concentration for which there is a stipulated chance (such as 95%) that something will be detected as a positive (i.e.; in this instance, there is a 5% chance of obtaining a false negative). See Detection Limit.
Limit of Quantitation (LOQ)	It is considered as the lower limit for which precise quantitative measurements can be made. A frequent rule used for the LOQ is that it is equal to the blank plus 10 times the standard deviation of the blank. Some consider this equivalent, or nearly equivalent to the PQL.
Machine Direction (MD)	In paper making, a screen (called a 'wire') passes under equipment (headbox) that dumps pulp onto it. Since this screen is moving, the fibers tend to orient in the direction the screen is running. This orientation is referred to as the machine direction. The perpendicular is the cross direction, or the direction across the machine.
May	Action, activity, or procedural step that is neither required nor prohibited. (EPA)
May not	Action, activity, or procedural step that is prohibited (EPA).
Matrix	The component or substrate which contains the analyte of interest. For purposes of batch determination, the following matrix types shall be used: Aqueous - Any aqueous sample not defined as drinking water or Saline/Estuarine water. Includes surface water, groundwater and effluents. Drinking water - Any aqueous sample that has been designated a potable or potential potable water source. Saline/Estuarine - Any aqueous sample from an ocean or estuary, or other salt water source. Non-aqueous liquid - Any organic liquid with <15% settleable solids. Biological Tissue: Any sample of a biological origin. Such samples shall be grouped according to origin.

	Paper or pulp. Solids - Includes soils, sediments, sludges and other matrices with >15% settleable solids. Chemical Waste - A product or by-product of an industrial process that results in a matrix not previously defined. Air Samples - Media used to retain the analyte of interest from an air sample such as sorbent tubes or summa canisters. Each medium shall be considered as a distinct matrix.
Matrix Spike (spiked sample, fortified sample)(MS)	A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to indicate the effect of the matrix on a method's recovery efficiency. Note that this is only an indicator. True positive or negative effect cannot really be determined for most materials.
Matrix Spike Duplicate (MSD)	A second replicate matrix spike which is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Measurand	Particular quantity that is subject to measurement. (ISO)
Measurement	A comparison process in which newly acquired quantitative information is compared with conventionally defined units.
Method	Denotes a set of written instructions specifying a testing procedure to be followed by an analyst or tester in order to obtain a numerical estimate of the concentration in a sample or value of a parameter of a sample Differentiated from an SOP in that the term is used for obtaining specified results, not general processes.
Method Blank	See Blank.
Method Detection Limit (MDL)	1. Ideally, this is the detection limit as it applies to the method. This is generally much larger than the IDL, but for some methods may be the same. 2. For many samples that fall under the Federal Clean Water Act or other regulatory systems, the procedures for developing MDL's are defined by statute. The regulatory MDL is not the true detection limit of a method. (The MDL is quite statistically and scientifically flawed.)
Method of Standards Addition	A technique wherein equal volumes of sample are added to a blank and a series (≥3) of standards. The blank and standards must also be equal in volume. The instrument response from each solution is plotted on a graph. The concentration on the graph when plotted back to zero response gives the concentration in the sample. The slope should not be more than 20 % off from a normal curve

	without sample. Also, the response must be linear and there must not be any spectral or background interferences. See EPA manual EPA-600/4-79-020 as revised March 1983, page METALS-12, section 8.5. The standards should be at 0.5, 1, and 1.5 times the measured native analyte concentration.)
Minimum Level (ML)	1. Equal 3.18 times the MDL, or 10 times the 's' in the MDL determination. 2. Current EPA guidelines takes this as the lower limit of the method for which regulatory enforcement is applied. 3. Under 40CFR430, App. A or 40CFR430.01: The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.
Minimum Measurement Level (MML)	Minimum measurement level is the concentration value used in compliance demonstration calculations when the method measures non-detect for a hazardous air pollutant (HAP) as per 40CFR63.457(c)(5)[air regulations].
Modulus of Elasticity	The ratio of linear 'engineering' stress (amount of stretch in paper) to the corresponding 'engineering' strain (pulling force or tension) below the proportional (elastic) limit in paper. Such a relationship in tension is called the Young's modulus. Below the proportional limit, the stress/strain relationship should be linear.
Modulus of Rupture	The maximum value of linear 'engineering' stress (amount of stretch in paper) at failure.
Must	Denotes a requirement that must be met (as opposed to 'shall').
Negative Control	Measures taken to ensure a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
NELAC	National Environmental Laboratory Accreditation Conference. A “voluntary” organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset and governing body of NELAP. NELAC itself does not certify.
NELAP	The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. NELAP does the certifying.
Non-volatile	Considered (in this laboratory) to be those components of a sample, such as pulp, left after oven-drying at 105°C. Generally, this is attributable to water, but other low molecular weight material can also be driven off.

Normal (N)	Gram molecular weight of solute divided by hydrogen equivalent of solute, per liter of solution
Opacity	The degree to which a sheet obscures a pattern beneath it.
Original Data	See Raw Data.
Original Record Book	A bound, paginated book used to record original data used for research purposes. Records so kept must follow Company ORB policies. Not to be confused with an ORF.
Original Record File	A file comprised of all data associated with an SR and assembled for archive, with associated archive retrieval number.
Performance Based Measurement System (PBMS)	A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate methods to meet those needs in a cost-effective manner.
Population (statistical)	The collection or set of all possible analytical or test results having a common observable characteristic.
Positive Control	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Practical Quantitation Limit (PQL)	An EPA term for the lower limit of quantification. It is considered as the lower limit for which precise quantitative measurements can be made. A frequent rule used for the PQL is that it is equal to 10 times the standard deviation of the blank. Generally viewed at 3 times the MDL. Viewed by some as the concentration level where results from 80% of the laboratories are within ± 40 % of the true value.
Prep(aration) Blank (PB)	See Blank.
Precision	A qualitative term to denote the scatter of results obtained under similar conditions. Precision is said to improve as the scatter among results becomes smaller. Also referred to as imprecision. Usually expressed as standard deviation, in either absolute or relative (%) terms. Variance or range is sometimes used, but is not desired in this laboratory.
Preservation	Physical measures applied and/or reagents added at the time of sample collection to maintain the (chemical) integrity of the sample. This can include maintenance at room temperature, refrigeration, protecting from light, acidification, and "fixing" a property or analyte (such as precipitating sulfide with zinc).

Proficiency Test Sample (PT)	A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits.
Proficiency Testing	Determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.
Proportional Limit	The greatest 'engineering' stress, which paper is capable of sustaining without any deviation from the proportionality of stress to strain (Hooke's law.) (Sort of the limit where the material ceases to be elastic and becomes inelastic or 'plastic'.
Protocol	A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed, such as an SOP, method, or regulation.
Quality Assurance(QA)	An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.
Quality Control (QC)	The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. A subset of QA.
Quality Control Sample	See Control.
Quantitation Limit (QL)	Convention takes this to be the lowest level for which a substance can be quantified at a specified level of confidence. Levels below the QL, but above the detection limit, can only be determined to be present or absent. Generally the QL is where the confidence limit is 40% or less. NELAC has defined this as three times the MDL. See 'PQL.' In reality, the QL is the maximum or minimum levels or concentrations of a substance that can be measured with the confidence level required by the data user.
Quantitative	Concentration measurement results that are above the specified quantitation limit and below the upper end of the calibration range of the measurement process.
Random Errors	These are indicated when repeated analyse of identical portions of a homogeneous sample does not give a series of identical results. The results differ among themselves and are, more or less, scattered about some value. They are termed random because the sign and magnitude of the error of any particular result vary at random, and cannot be predicted exactly.

Range	Generally, the difference between the minimum and the maximum of a set of values. Sometimes expressed as the upper and lower limits of that range.
Raw Data	Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, or notes that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. Exact copies of raw data that have been prepared (e.g., tapes transcribed verbatim, dated and verified accurate by signature) may be submitted in specified cases, but are not considered to be raw data.
Reagent Blank	See Blank
(Analytical) Reagent (AR) Grade	Designation given by the American Chemical Society for certain chemical reagents and solvents meeting its stipulated specifications.
(Pure) Reagent Water	Water in which no target analytes or interferences are detected as required by the analytical method, meeting ASTM Type I or Type II criteria, or better.
(Analytical) Recovery	An estimate usually expressed in percent, of an analytical or testing result compared to a true or reference value for an analyte or substance. For example, an analytical recovery of 95% for compound X means that the result was 95% of the true or reference value for X in the sample. It is a direct indication of analytical performance.
Reference Material (RM)	A material or substance of which one or more properties are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. Generally, in this lab, RMs are used on an occasional basis, not as control samples.
Reference Standard	A standard, generally of the highest quality of metrology available, at a given location, from which measurements are derived. It may be the same as a RM or SRM in some instances.
Reflectance	The ratio of reflected to incident radiation (i.e.; light.).
Reflectivity	The reflectance of a material measured in a layer so thick that an increase in thickness does not alter reflectance.
Relative Percent Difference (RPD)	The range, or difference between, two values divided by the mean of the two values multiplied by 100.

Relative Standard Deviation (RSD)	Also known as the coefficient of variation (COV). The relative percent standard deviation relative to the mean. Equal to the standard deviation (s) divided by the mean times 100 . It is calculated as $100s/\bar{x}$. It is sometimes referred to as a percent relative standard deviation, although the percent is redundant.
Repeatability (TAPPI)	The limit within which the agreement of two test results may be expected to be 95 % of the time. 2.77 (for 95%) times the standard deviation, s_r, of results obtained by the same operator using the same instrument in immediately successive measurements. (see TAPPI T1206.) The term is a little restrictive for general usage and its use is discouraged within this laboratory. TAPPI Repeatability = 2.77 (s_r). In TAPPI round robins, s_r = pooled standard deviation, s_p, of the replicated test values which are used to give a result. (see TAPPI T 1206.) Within the laboratory, an estimate of s_r may be determined by using an estimate of the standard deviation, s_e. In this case $s_r = s_e/(m)^{½}$,) where m = number of replicate values within a single test result and s_e is the standard deviation of the number of test results.
Replicate (Analyses)	The measurements of parameter performed identically on two or more sub-samples of the same sample within a short time interval. The results from replicate analyses may be used to evaluate analytical or measurement precision. Not intended to indicate precision of sampling, preservation, or storage internal to the laboratory.
Reproducibility (TAPPI)	The limit within which the agreement of two test results from different qualified laboratories may be expected be 95 % of the time. The term is a little restrictive for general usage and its use is discouraged within this laboratory. TAPPI Reproducibility = 2.77 (s_R) where s_R is the standard deviation of the results of a test from each laboratory participating in the study.
Scattering Coefficient	Rate of increase of reflectance with thickness
Selectivity	In analytical chemistry, it is the capability of a method or instrument to respond to a target substance or constituent in the presence of non-target substances.
Sensitivity	The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. In some cases, such as atomic absorption, it is defined by convention.
Shall	This denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This <u>does not prohibit</u> the use of <u>alternative approaches</u> or methods for implementing the specification so long as the requirement is fulfilled (as opposed to "must"). (ANSI)

Should	Action, activity, or procedural step that is suggested but not required. (EPA)
Significant Figure	Generally the number of digits necessary to express results consistent with the measured precision and accuracy. Customary convention is all digits that are 'known' plus the first uncertain digit.
Spike	The addition of a known amount of analyte to a sample to judge, from the analytical percent recovery, whether there is any bias or lack of precision because of interference present in the sample. Also referred to as fortification of the sample.
Spike cont.	Analytical (or analysis) Spike – A spike added to a sample just before running on an instrument. It is used to ascertain problems at the instrument level, as opposed to the preparation level. A.k.a., "A-spike." Field Spike – Added to samples in the field to check recovery of a parameter from the process of sampling, preservation, and transportation. Method Spike – Added to samples at the beginning of an analysis to check recovery of the entire analysis procedure, including digestion or extraction.
Standard	A substance of known value used for standardizing an instrument or procedure.
Standardize	The process of establishing a response that can be quantified to know value. Generally, an instrument response (absorbance, emission, GC column response) to a known concentration or amount of material for purpose of making comparable measurements. Frequently and improperly confused with calibration.
Standard Deviation (sd)	A constant which describes the width of the normal distribution. For a sample of n results taken from a population it is calculated as follows: $s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{(n-1)}} = \sqrt{\frac{\sum x_i^2 - (\sum x_i)^2 / n}{(n-1)}}$
Standard Operating Procedure (SOP)	A detailed written description of a method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the process for performing certain routine or repetitive tasks. In this laboratory it is usually used for general processes and instrument operations, not methods (for example, preparing glassware, operating an AA, etc). It is often incorporated by reference in a method.
Standard Reference Material(SRM)	A certified reference material, such as one issued by the National Institute of Standards and Technology (NIST), characterized for absolute content or characteristic, independent of analytical or testing method.
Standard uncertainty (ISO)	The standard deviation of a set of values, or an estimated derived standard deviation where original information is not present.

Statistical Sample	The results of one or more determinations (taken from the population of all possible results).
Statistics	Certain single values computed from the results, which characterize certain properties of the results. Each statistic has its own frequency distribution that is defined by a particular mathematical function. Examples of statistics are the mean, $\bar{x}$, standard deviation, s, and the frequently used t- and F- statistics.
Support Equipment	Devices that may not be the actual test instruments, but are necessary to support laboratory operations. These may include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors,) and volumetric dispensing devices.
Surrogate Standard	A type of check standard, which is added to every sample, in a known amount, at the start of processing for quality control purposes. The surrogate is not one of the target compounds for the analysis, but should have analytical properties similar to those compounds. The surrogate compounds are not expected to be present in environmental samples.
Systematic Errors	These are indicated by a tendency of results to be greater or smaller than the true value. Usually bias can be considered equivalent to systematic error.
Tensile Strength	Maximum 'engineering' stress paper is capable of sustaining, i.e., the maximum load of a tension test carried to rupture.
Tentatively Identified Compounds	Non-targeted substances tentatively ‘identified’ on a GC/MS chromatogram because their mass spectra correspond to spectra found in a library. They are not quantified with standards; hence ‘concentration’ is arbitrarily determined by comparison with responses to other substances.
Testing System	Denotes a combination of tester, test method, equipment, standards, laboratory facilities, and any other components involved in carrying out an test procedure.
Test Method	See Method.
Test Result	Denotes a numerical estimate of the value or magnitude of a property, or concentration of a determinant (analyte,) in a sample, and is obtained by carrying out, once, the procedure specified in a test method. Note that a method may specify analysis or testing of more than one portion of a sample in order to produce one test result. The result can also be thought of as the final value reported to the user.
Test Response	A numerical observation which is obtained when a sample or portion of a sample is presented to a measurement system or sub-system. The magnitude of the response is related to the magnitude of property being measured.

Test Sensitivity/ Power	The minimum significant difference (MSD) between the control and test concentration that is statistically significant. It is dependent on the number of replicates per concentration, the selected significance level, and the type of statistical analysis.
Tolerance Chart	A chart in which the plotted quality control data is assessed via a tolerance level (e.g. ± 10 % of a mean.) This type of chart is based on the precision level judged acceptable to meet overall quality/data use requirements, instead of statistical acceptance criteria (e.g. ± 3 s) where 's' is the standard deviation. NOTE: Not to be confused with tolerance used in a statistical tolerance interval. (See QA Advisor for more information.)
Traceability	The property or a result of a measurement, or property of a material, or property of an apparatus whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.
Traceable	Traceability of measurements (&/or calibrations) (in SI units) to national or international standards of measurement or international standard reference materials, e.g.; NIST, can be demonstrated. The source of the standard shall provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. Standard suppliers of chemical standards (APG, AccuStandards, ULTRA) certified by the NVLAP-NIST proficiency program are considered competent for specified parameters. (Certificates from private vendors should meet ASTM D6362.)
Warning Limit (WL)	A type of control limit which is specified by a value on a control chart, usually ±2s distant from the expected value, where 's' is the standard deviation. Action is required when results fall outside the warning limits too frequently. A single value outside the warning limit does not require action, but should alert one to a possible problem.
Wire side	In making of paper, the pulp is normally formed on a screen (called a 'wire').When this is done, mini-rectangles form on the sheet caused by the pulp sagging though the holes in the screen. The opposite side of the sheet is commonly referred to as the 'felt' side.
Verification	The confirmation by examination, and provision of evidence, that specified requirements are met. The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between measured values and corresponding known values are consistently smaller than the maximum allowable error. This error may be defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

Validation	The process of substantiating specified performance criteria.
Young's Modulus	See Modulus of Elasticity.

Qualifiers – Analytical Testing Only

Qualifiers (flags) for Organic Analysis Reports

A	For tentatively identified compounds that are suspected to be aldol-condensation products.
B	Indicates that the analyte is detected in the associated blank as well as the sample.
C	Used for pesticide results that have been confirmed by GC/MS.
D	For identifying all compounds identified in an analysis at a secondary dilution. Used to alert data user that any discrepancies between the concentrations reported in the two runs may be because of dilution errors.
E	Used for compounds whose concentration exceeds the calibration range of the instrument.
J	Used to indicate the reported value is an estimated value. Used either when 1) estimating a concentration for a tentatively identified compound or when 2) the data indicate the presence of a compound, but the results are less than the sample quantitation limit and are greater than zero. Sometimes used when the sample does not meet requirements, such as out of holding time.
N	Indicates presumptive evidence of a compound. Only used for tentatively identified compounds where the identification is based on a mass spectral library search.
P	Used for pesticide/Aroclor target analyte when there is greater than 25 % difference for the detected concentrations between two different GC columns. The lower of the two results is reported.
R	Quality control indicates datum is not usable.

Q	No analytical result
U	Indicates that the compound was analyzed for, but not detected at, the reported concentration. The sample quantitation limit, corrected for dilution and percent moisture, is reported.
X Y, Z	1. For some labs, used when it is assigned by the computer when the operator has manually adjusted the program. There is no significance on the datum itself. 2. Used to indicate reference to footnotes. Y and Z sometimes used if there is more than one X.

Qualifiers (flags) for Inorganic Analysis Reports

B	Value for the analyte is below the reporting limit. (Generally < CRDL.)
E	Value reported is an estimate, generally attributable to interference.
M	Indicates injection precision not met.
N	Indicates spiked sample not within control limits.
R	Quality control indicates datum is not usable.
S	Indicates the reported value determined by Method of Standards Additions. Not to be used with 'W' and '+'.
U	Indicates that the compound was analyzed for but not detected at the reported concentration. The sample quantitation limit, corrected for dilution and percent moisture, is reported.
W	Post-digestion spike for GFAA analysis is out of control limits (85-115%), while sample absorbance < 50 % of the spike absorbance. Not to be used with 'S' and '+'.
Z	No analytical result.
1.	See E.
*	Duplicate analysis is not within control limits.
+	Correlation coefficient for the MSA < 0.995. Not to be used with 'S' and 'W'.

	Method qualifiers
A	Flame AA.
AS	Semi-automated spectrophotometric.
AV	Automated Cold Vapor AA.
C	Manual spectrophotometric.
CV	Manual Cold Vapor AA.
F	Furnace AA.
NR	Analyte is not required to be analyzed.
P	ICP
T	Titrimetric

Index

Chapter and section location listed plus (page)

-A-
A&T Chemical Hygiene Plan
(see also chemical hygiene plan) 9.4 (p19)
absolute magnitude glossary (p36)
accuracy 5.3 (p12)
alarms 3.5 (p 6)
analysis spike 10.7.2 (p23)
analyte of interest. 5.7 (p12)
analytical standard 9.3 (p19)
APG Round Robin 4.1.1 (p 8)
AQ O-DLYCHK 8.5.2, 8.5.3 (p17)
AQ O-PIPCAL 8.5.4 (p17)
Attorney-client
privileged 15.6.3 (p33)
-B-
balances, analytical 8.5.3 (p17)
batch 10.1 (p20), 11.1 (p24)
blank 10.5.2 (p22)
blank matrix 10.6.2 (p22)
blind sample 10.5.1 (p22)
blue ink 14.8 (p31)
BOD incubators 8.5.2 (p17)
-C-
class A burettes 8.5.4 (p17)
calibration 8.3.1 (p16), 10.4.1 (p21)
calibration certificates 8.3.3 (p16), 8.6.1 (p18)
calibration criterion 10.2.4 (p21)
calibration curve(s) 8.4.2 (p17), 10.4.3 (p21), 14.9 (p31)
calibration levels 10.2.3 (p21)
calibration log 8.5.1 (p17)
calibration records 8.3.3 (p16)
calibration status,
current 8.3.2 (p16)
calibration
verification(s) 10.4.2 (p21), 10.4.3 (p21), 10.6.1 (p22)
calibration, equipment 8.6.2 (p18)

Index

Chapter and section location listed plus (page)

calibrations, multiple
(more than two) 14.14 (p31)
Certificate of Analysis 9.2 (p19)
chain-of-custody 4.5.6 (p10)
Chemical Hygiene Plan
(see also A&T Chemical Hygiene Plan)
3.3 (p 6), 3.7 (p7), 4.7.1 (p11)
chemical preservation 4.5.4 (p10)
chemical standards 8.4.1 (p17), 9.1 (p19)
class A burettes
(see burrettes)
class A glassware
(see glassware)
class A non-glassware
(see non-glassware)
computer manipulation 14.14 (p31)
confidence limits 13.15 (p28)
contaminants 5.7 (p12)
continuing calibration
verifications 8.4.3 (p17)
continuing verification
standard 10.4.1 (p21)
continuous improvement 2.4 (p 5)
Control Center 3.2 (p 6), 3.5(p 6)
control chart(s) 11.3 (p24), 11.4 (p24), 11.5.1 (p24)
copier data 14.8 (p31)
correction factor 8.3.4 (p16)
custom testing 7.4 (p14)
customer satisfaction 1.2 (p 3), 2.3 (p 4) , 2.4 (p 5)
customer's request. 5.1 (p12), 5.8 (p12)
-D-
detection limit(s) 10.3.1 (p21), 12.1 (p25), 13.12 (p28), 13.13 (p28)
detection, limits of 5.6 (p12)
disposal of chemicals
and reagents 9.4 (p19)
Dixon's "test" procedure 12.4.1 (p25)
-E-
electronic data files 15.5.3 (p32)

Index

Chapter and section location listed plus (page)

electronic transmission 15.5 (p32)
end-use needs 5.1 (p12)
equipment on loan 8.1 (p15)
equipment or calibration log 8.3.4 (p16)
equipment, new 8.2.3 (p16)
equipment, WATS spreadsheet 8.1.2 (p15)
-F-
facilities and housekeeping services 3.7 (p 7)
fails calibration/ verification 8.2.2 (p16)
field and trip blanks 10.5.3 (p22)
field blanks 12.2 (p25)
field spike 10.7.2 (p23)
-G-
glass micro-liter syringes 8.5.5 (p18)
glassware, class A 8.5.5
glassware, non-class A 8.5.6 (p18)
glassware, volumetric 8.5.5 (p18)
-H-
hand-altered material 14.4 (p30)
historical reconstruction 14.1 (p30)
historical requirements 5.8 (p12)
human factors 2.1 (p 4)
humidity 3.2 (p 6)
-I-
instruction and maintenance manuals 8.1.5 (p15)
instrument blanks 10.5.2 (p
instrument lab books 8.1.3 (p15)
instrument limitations 12.1 (p25)
instrument logbooks 8.1.4 (p15)
instrument peaks 14.1 (p30)
ISO 9001:2000 standard 1.4 (p 3), 2.4 (p 5)

Index

Chapter and section location listed plus (page)

-J,K,L-
lab control spiked sample (LCS) 10.6.2 (p22)
lab control spikes 10.6.1 (p22)
Laboratory Policies and Guidelines 8.1 (p15)
laboratory refrigerators and freezers 8.5.2 (p17)
limiting factor 13.7 (p26)
LIMS 14.6 (p31)
logbook(s) 8.2.2 (p16), 8.2.3 (p16), 8.5.2 (p17), 9.3 (p19)
-M-
maintenance log 8.5.1 (p17)
matrix 5.4 (p12)
Matrix spike (method spike) 10.7.2 (p23)
matrix spike duplicates 10.8.1 (p23)
MDL (Method Detection Limit) 13.13 (p28)
mean 11.5.1 (p24)
measured precision and accuracy 13.2 (p26)
mechanical volumetric dispensing devices 8.5.4 (p17)
method blank 10.5.1 (p22)
Millipore systems 8.5.2 (p17)
-N-
nonconformances 7.2 (p14)
non-glassware, class A 8.5.5 (p18)
non-standard testing 7.4 (p14)
-O-
official reports 15.5.2 (p22)
opinions and interpretations 15.3 (p32)
original data 14.8 (p31)
original documents and data 14.3 (p30)
outlier determination 12.4.1 (p25)

WATS Laboratory Policies and Guidelines
Index

Chapter and section location listed plus (page)

Outsourced Services 3.7 (p 7)
ovens 8.5.2 (p17)
-P-
performance audits 4.3.2 (p 9)
precision 5.4 (p12)
preventative maintenance program 8.2.1 (p16)
print-only PDF 15.5.2 (p32)
pulp and paper testing 1.4 (p 3)
-Q-
QC Control charts 11.2 (p24)
QMS (see quality management system)
quality guidelines 1.4 (p 3)
quality management system (QMS) 1.1 (p 3), 2.2 (p4), 2.3 (p4)
quality product 1.2 (p 3)
-R-
r acronyms (p36)
reagent, grade of the 9.1 (p19)
reagent, quality of a 9.3 (p19)
reagents 9.1 (p19)
reference materials 10.6.1 (p22), 10.6.3 (p22)
reference standards 8.6.5 (p18)
regulations 5.2 (12), 7.3 (p14)
regulatory procedures 7.1 (p14)
repeatability 7.2 (p14)
replicates 11.4 (p24)
reporting limit 14.9 (p31)
reports with errors 15.4 (p32)
restrict access 3.6 (p 6)
review of client requests (contracts) 6.2 (p13)
'Revised Report' 15.4.2 (p32)
room temperature 3.2 (p 6)
round robin testing 2.4 (p 5)
rounding off 13.3 (p26)
-S-
safety 5.1 (p12), 5.8 (p12)

Index

Chapter and section location listed plus (page)

Sample Custody Log 4.7.1 (p11)
sample disposal 4.7.1 (p11)
sample handling 4.3.1 (p 8)
Sample Management Office (SMO) 4.1.1 (p 8), 4.1.2 (p 8)
sample matrix 5.7 (p12)
Sample Receipt Protocol 4.5.2 (p10)
sample submittal 4.1 (p 8)
sampling 4.3.1 (p 8)
service request data sheets 9.3 (p19)
significant digit(s) 13.8 (p27), 13.9 (p27), 13.12 (p28), 13.14 (p28)
significant figure(s) 13.1 (p26), 13.2 (p26), 13.6 (p26), 13.7 (26), 13.9 (p27), 13.11 (p28), 13.13 (28)
slopes 14.1 (p30)
spike recovery 10.7.1 (p22)
spike(s) 10.7.1 (p22), 11.4 (p24)
spike, surrogate 10.7.2 (p22)
standard calibration curves 8.4.3 (p17)
standard deviation 13.15 (p28)
standard operating procedures 2.4 (p 5), 3.7 (p 7)
standard reference 10.7.1 (p22)
standardization curves 8.4.2 (p17)
standardization of the instrument 10.2.1 (p20)
standards 11.4 (p24)
subcontract(ed)(ing) 6.1 (p13), 6.3 (p13), 6.4 (p13), 6.5 (p13)
support equipment 8.5.1 (p17)
-T-
“T” table 12.4.1 (p25)
temperature requirements 4.5.3(p10)
temporary or mobile facility 2.5 (p 5)
test methods 2.4 (p 5), 3.7 (p 7)

WATS Laboratory Policies and Guidelines
Index

Chapter and section location listed plus (page)

Total Quality Management (TQM) 2.3 (p 4)
total quality management process 1.1 (p 3)
traceability 9.2 (p19), 9.3 (p19)
Traceability of Calibration 8.5.1 (p17)
traceability of measurements 8.6.1 (p18)
traceable (ability) to national standards 8.6.3 (p18), 8.6.4 (p18)
training records 2.4 (p 5)
trip blanks 12.2 (p 25)
-U,V-
unethical or illegal behavior or actions 14.12 (p 31)
validity of the tests 7.3 (p14)
verification and validation of measurements 8.6.2 (p18)
-W-
warning limits 11.5.1 (p24)
water stills 8.5.2 (p17)
WATS (Weyerhaeuser Analysis and Testing Services) 1.1 (p 3)
WATS Laboratory Policies and Guidelines 1.4 (p 3), 2.3 (p 4), 2.4 (p 5)
WATS Quality Assurance Manual 3.7 (p 7)
WATS quality management system 1.2 (p 3), 2.3 (p 4)
WATS Quality Manual 1.4 (p 3), 2.4 (p 5), 8.1 (p15)
Weyerhaeuser Analysis and Testing Services (WATS) 2.1 (p 4)
Weyerhaeuser's Chemical Management Department 9.4 (p19)
witnessing and dating 14.5 (p30)
-X,Y,Z-
yearly quality objectives 1.1 (p 3), 2.4 (p 5)