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Subject:
Multi-Area Quality Assurance Project Plan
Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site

Date:
June 29, 2007

Contact:
Michael J. Erickson

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Our ref:
B0064524 and B0064530

Dear Mr. Saric and Mr. Borries:

On behalf of the Kalamazoo River Study Group (KRSG), please find enclosed the Multi-Area Quality Assurance Project Plan (QAPP). This Multi-Area QAPP has been prepared following the Universal Federal Policy (UFP) QAPP guidance according to the requirement of the Administrative Settlement Agreement and Order on Consent (AOC) for Remedial Investigation and Feasibility Study with the U.S. Environmental Protection Agency (USEPA) (Comprehensive Environmental Response, Compensation, and Liability Act [CERCLA] Docket No. V-W-07-C-864 [SRI/FS AOC]). As agreed upon with USEPA, this Multi-Area QAPP addresses environmental monitoring to be conducted under the SRI/FS AOC as well as the former Plainwell Impoundment Time Critical Removal Action [TCRA] AOC (CERCLA Docket No. V-W-07-C-863 [TCRA AOC]).

Each of the enclosed packets contains replacement cover, spine and pages for the draft version you received in April as well as a CD with a portable document format (PDF) version of the entire final document. Additional copies are being sent on CD as presented in the cc list below.

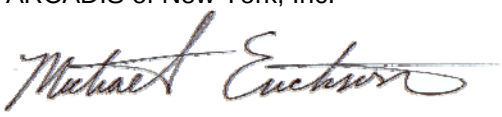
On May 23, the USEPA notified the KRSG of approval of the *Multi-Area Quality Assurance Project Plan* (QAPP) provided revisions were made to the draft per the letter notification of approval. The USEPA's comments provided in that letter were incorporated in preparing this final QAPP.

Please return a signed signature page to me at your earliest convenience. A return addressed, postage-paid envelope is included for your convenience.

If you have any questions, please do not hesitate to contact us.

Sincerely,

ARCADIS of New York, Inc.



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Multi-Area Quality Assurance Project Plan

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Supplemental Remedial Investigations/
Feasibility Studies

Multi-Area Quality Assurance Project Plan (Rev. 0.0)

Kalamazoo River Study Group

June 2007

ARCADIS BBL

Multi-Area Quality Assurance Project Plan (Rev. 0.0)

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

Supplemental Remedial Investigations/Feasibility Studies

Prepared for:
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June 2007

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Attachments

Attachment 1-1 Chain of Custody

Attachment 1-2 Laboratory Certifications

Attachment 1-3 Laboratory Quality Assurance Management Plans and SOPs (included on CD only)

AOC	Administrative Order of Consent
ARAR	applicable or relevant and appropriate requirement
ASTM	American Society of Testing and Materials
AVS	acid volatile sulfides
AVS/SEM	acid volatile sulfides/simultaneously extracted metals
BBEPC	Blasland & Bouck Engineers, P.C.
BBL	Blasland, Bouck & Lee, Inc.
BERA	Baseline Ecological Risk Assessment
BFB	bromofluorobenzene
ºC	degrees Celsius
CA	corrective action
CAL	calibration
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CF	calibration factor
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COB	close of business
COC	chain-of-custody

CQA	construction quality assurance
CSM	Conceptual Site Model
CSV	comma-separated value
Design Report	*Time-Critical Removal Action Design Report for the former Plainwell Impoundment*
DFTPP	decafluorotriphenylphosphine
DO	dissolved oxygen
DQI	data quality indicator
DQO	data quality objective
DRO	diesel-range organic
ECO RA	ecological risk assessment
EDD	electronic data deliverable
ERA	Ecological Risk Assessment
FID	flame ionization detector
FS	Feasibility Study
FSP	Field Sampling Plan
GC/ECD	gas chromatography/electron capture detectors
GC/MS	gas chromatography/mass spectrometry
GIS	Geographic Information System
GPS	Global Positioning System

GRO	gasoline-range organic
HHRA	Human Health Risk Assessment
HSP	Health and Safety Plan
ICP-AES	inductively coupled plasma – atomic emission spectrometry
ICV	initial calibration verification
ID	identification
KAR	KAR Laboratories, Inc.
KRSG	Kalamazoo River Study Group
L	liter
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
MDEQ	Michigan Department of Environmental Quality
MDL	method detection limit
MDNR	Michigan Department of Natural Resources
mg/kg	milligram per kilogram
mg/L	milligram per liter
mL	milliliter
MS	matrix spike
MSD	matrix spike duplicate
MSSL	Medium-Specific Screening Level

NA	Not Applicable/Available
µg/L	microgram per liter
NCP	National Contingency Plan
NEIC	National Enforcement Investigations Center
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Science and Technology
NOAA	National Oceanic and Atmospheric Administration
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste and Emergency Response
OU	operable unit
PAH	polycyclic aromatic hydrocarbon
PCBs	polychlorinated biphenyls
ppm	part per million
PQO	project quality objectives
QA	quality assurance
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QA/QC	quality assurance/quality control
QC	quality control
RA	risk assessment

RBC	Risk-Based Concentration
RCRA	Resource Conservation and Recovery Act
RF	response factor
RI/FS	Remedial Investigation/Feasibility Study
RL	reporting limit
RPD	relative percent difference
RPM	Remedial Project Manager
RRD	Remediation and Redevelopment Division
RRF	relative response factor
RSD	relative standard deviation
SAP	Sampling and Analysis Plan
SDG	sample delivery group
SEM	simultaneously extracted metals
Site	Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site
SOM	Superfund Organic Method
SOP	Standard Operating Procedure
SOW	Statement of Work
SRI	Supplemental Remedial Investigation
STL	Severn Trent Laboratories, Inc.
SVOCs	semi-volatile organic compounds

TBD	to be determined
TCP	Traffic Control Plan
TCRA	Time-Critical Removal Action
TCL/TAL	Target Compound List/Target Analyte List
TDL	target detection limit
TDS	total dissolved solids
TOC	total organic carbon
TPH	total petroleum hydrocarbon
TSS	total suspended solids
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service
USGS	United States Geological Survey
VOC	volatile organic compound
VTSR	validated time of sample receipt
Work Plan	*Supplemental Remedial Investigation and Feasibility Study Work Plan*
%D	percent difference
%R	percent recovery

1. Introduction

On February 21, 2007 Georgia-Pacific Corporation (Georgia-Pacific) and Millennium Holdings, LLC—collectively referred to as the Kalamazoo River Study Group, or KRSG—voluntarily entered into two separate agreements (Administrative Settlement Agreements and Orders on Consent [AOCs]) with the U.S. Environmental Protection Agency (USEPA) that will govern the majority of work from this point forward at the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site (Site), located in Kalamazoo and Allegan counties in southwest Michigan (Figure 1-1). One agreement describes a series of supplemental remedial investigations and feasibility studies (SRI/FSs) that will be carried out over the next several years in Operable Unit 5 (OU5) of the Site, which encompasses approximately 80 miles of the Kalamazoo River, including a stretch of Portage Creek from Cork Street to its confluence with the Kalamazoo River (SRI/FS AOC; Comprehensive Environmental Response, Compensation and Liability Act [CERCLA] Docket No. V-W-07-C-864; USEPA 2007a). The second AOC describes the requirements for a Time-Critical Removal Action (TCRA) in the former Plainwell Impoundment (Figure 1-2) of the Kalamazoo River (TCRA AOC; CERCLA Docket No. V-W-07-C-863; USEPA 2007b). Work on the TCRA began in the spring of 2007, and removal activities and initial planting of vegetation are expected to be complete in the spring of 2009.

Both the SRI/FS AOC and TCRA AOC require development and submission of a Quality Assurance Project Plan (QAPP) to describe sample analysis and data handling requirements for samples collected during work at the Site.

This *Multi-Area Quality Assurance Project Plan* (Multi-Area QAPP) has been developed to meet the requirements of the activities to be performed for the SRI/FSs and the TCRA in the former Plainwell Impoundment. It provides details on the sampling and analytical methods and procedures that will be used during investigations at the Site and for monitoring during and after the TCRA construction activities. This Multi-Area QAPP, which is designed to be used in conjunction with other supporting documents, such as the *Multi-Area Health and Safety Plan* (Multi-Area HSP; ARCADIS BBL 2007d) and *Multi-Area Field Sampling Plan* (Multi-Area FSP; ARCADIS BBL 2007e), will be modified as necessary as the sampling programs develop.

1.1 Development of the Multi-Area QAPP

As described on USEPA's Quality System Website (http://www.epa.gov/QUALITY/qapps.html), a QAPP is to document the planning, implementation and assessment procedures for a particular project, as well as any specific quality assurance and quality control activities. It is to integrate all the technical and quality aspects of the project to provide a "blueprint" for obtaining

the type and quality of environmental data and information needed for a specific decision or use.

In March 2005, the Intergovernmental Data Quality Task Force developed a policy and guidance document to fulfill the project-specific requirements of Part B of ANSI/ASCQ E4, to ensure that Federal departments and agencies will produce consistent QAPPs that reflect a systematic planning approach to collection and use of environmental data. That guidance document—the *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP) Manual V1, March 2005 (USEPA 2005a)—was followed in the development of this Multi-Area QAPP, per requirement of Section 1.2.1.2 of the SRI/FS AOC Scope of Work. Additionally, the provisions of this Multi-Area QAPP were reviewed against the Minimum QA/QC Activities identified in the guidance document *Uniform Federal Policy for Quality Assurance Project Plans. Part 2B, Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities* V1, March 2005 (USEPA 2005a).

1.2 Project Setting

The Site is located in Kalamazoo and Allegan counties, and encompasses the Kalamazoo River from Morrow Dam to Lake Michigan and Portage Creek from Cork Street to the confluence with the Kalamazoo River (Figure 1-1). The former Plainwell Impoundment—which is part of OU5 and is the target area for the TCRA—is located in Gun Plain and Otsego Townships, downstream of Plainwell, Michigan (Figure 1-2). It is roughly bounded on its upstream (or southeastern) end by the Main Street Bridge in Plainwell, and on its downstream (or northwestern) end by the Plainwell Dam.

1.3 Applicability of the Multi-Area QAPP

In the Statement of Work (SOW) attached to the SRI/FS AOC, specific requirements for the SRI/FS activities are described, and seven distinct Areas within OU5 are defined. The SOW governs work in the following seven Areas of OU5:

- Area 1: Morrow Dam to Plainwell Dam, which includes a stretch of Portage Creek from Alcott Street to its confluence with the Kalamazoo River
- Area 2: Plainwell Dam to Otsego City Dam
- Area 3: Otsego City Dam to Otsego Dam
- Area 4: Otsego Dam to Trowbridge Dam

- Area 5: Trowbridge Dam to the Allegan City Dam
- Area 6: Allegan City Dam to Lake Allegan Dam
- Area 7: Lake Allegan Dam to Lake Michigan

The SOW also requires investigation of former paper mill properties (Mill Properties) to determine whether each Mill Property is a source of potential source of polychlorinated biphenyls (PCBs) to the Site. The Mill Properties are as follows:

- Former Bryant Mill
- Former King Mill and the King Street Storm Sewer area
- Former Monarch Mill
- Georgia-Pacific Corporation Kalamazoo Mill and former Hawthorne Mill (These mills are part of OU6[1] of the Site—only the mill investigations will proceed until the terms of the SRI/FS AOC and SOW. The rest of the work at OU6 will continue under separate, pre-existing agreements.)

Although the Multi-Area QAPP was developed primarily for work under the Areas and Mill Properties presented above and the activities associated with the TCRA, it is applicable to future work in other areas as well and will be updated as needed to cover future activities.

1.4 SRI/FS Activities

As described in the SOW included as Attachment A to the SRI/FS AOC, an objective of the SRI/FS is to evaluate the nature and extent of hazardous substances, pollutants or contaminants at and/or from the areas defined in the AOC; and, to assess the potential risks to human health and the environment posed by these hazardous substances, pollutants or contaminants, and alternatives for addressing the impacts to human health and the environment at each Area.

[1] OU6 is defined as the Georgia-Pacific Corporation Kalamazoo Mill Lagoons.

The first phase of the SRI/FS work will be carried out in Area 1; the portion of the Kalamazoo River between Morrow Dam and the Plainwell Dam, and a section of Portage Creek from Alcott Street to the confluence with the Kalamazoo River. The activities associated with this work are described in the *Supplemental Remedial Investigation/Feasibility Study Work Plan – Morrow Dam to Plainwell* (Area 1 SRI/FS Work Plan; ARCADIS BBL 2007a). The Area 1 SRI/FS Work Plan was approved by USEPA and is attached to the SRI/FS AOC.

1.5 Former Plainwell Impoundment TCRA

Through agreements with the USEPA, a TCRA will be conducted to address PCB contamination targeted sediments, river bank soils and floodplain soils within the former Plainwell Impoundment . Environmental monitoring, which is covered by this Multi-Area QAPP, will be conducted within and downstream of the former Plainwell Impoundment during and following TCRA construction activities. The TCRA and associated monitoring is described in the *Former Plainwell Impoundment Time-Critical Removal Action Design Report* (TCRA Design Report; ARCADIS BBL 2007b), which was approved by USEPA in February 2007. The Design Report is included as Appendix 4 to the TCRA AOC.

2. Document Overview

The UFP-QAPP guidance (USEPA 2005a) includes a series of 37 worksheets that can be used to present the critical information required in a QAPP. Since the worksheets were designed to "ensure consistent content and presentation of information in a project-specific QAPP... and streamline the review of QAPPs by regulators and others" (USEPA 2005a), this Multi-Area QAPP has been developed using these worksheets, which provide information associated with four key topics:

- Project Management and Objectives
- Measurement and Data Acquisition
- Assessment and Oversight
- Data Review

A series of attachments is also included with this Multi-Area QAPP. These attachments include

- chain of custody forms
- Laboratory Quality Assurance Plans and SOPs
- laboratory certifications.

When elements required by the UFP-QAPP are present in other documents (e.g., SOPs), careful cross-referencing of these other documents can be used in lieu of repeating information (USEPA 2005a). Following this guidance, this Multi-Area QAPP provides certain relevant information by reference to the Area 1 SRI/FS Work Plan (ARCADIS BBL 2007a), the TCRA Design Report (ARCADIS BBL 2007b), the Master Schedule (ARCADIS BBL 2007c), the Multi-Area Health and Safety Plan (HSP) (ARCADIS BBL 2007d), the Multi-Area Field Sampling Plan (FSP) (ARCADIS BBL 2007e), and the Data Management Plan (ARCADIS BBL 2007F). All of these planning documents are companion documents to the Multi-Area QAPP.

3. References

ARCADIS BBL. 2007a. *Supplemental Remedial Investigation/Feasibility Study Work Plan – Morrow Dam to Plainwell* (Area 1 SRI/FS Work Plan). February 2007.

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USEPA. 2005b. Intergovernmental Data Quality Task Force. *Uniform Federal Policy for Quality Assurance Project Plans. Part 2B, Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities.* Version 1, March 2005. Publication Numbers: USEPA: EPA-505-B-04-900B, Department of Defense: DTIC ADA 426957. http://www.epa.gov/swerffrr/pdf/qaqc_v1_0305.pdf.

USEPA. 2007a. Administrative Settlement Agreement and Order on Consent (SRI/FS AOC), CERCLA Docket No. V-W-07-C-864. United States Environmental Protection Agency Region 5. Effective February 21, 2007.

USEPA. 2007b. Administrative Settlement Agreement and Order on Consent for Removal Action (TCRA AOC), CERCLA Docket No. V-W-07-C-863. United States Environmental Protection Agency Region 5. Effective February 21, 2007.

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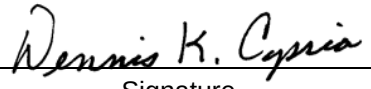
WORKSHEETS

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) three miles of Portage Creek and the Kalamazoo River from the City of Kalamazoo to Lake Michigan
Document Title:	*Kalamazoo River Study Group, Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site, Multi-Area Quality Assurance Project Plan – 2007*
Lead Organization:	USEPA Region 5
Preparer's Name and Organizational Affiliation:	Dennis K. Capria, ARCADIS BBL
Preparer's Contact Information:	6723 Towpath Rd., P.O. Box 66, Syracuse, NY 13214-0066, 315.671.9299, Dennis.Capria@ARCADIS-US.com
Preparation Date:	April 2007
Investigative Organization's Project Manager (SRI/FS Project Coordinator)	 Signature Michael J. Erickson, P.E., ARCADIS BBL
Investigative Organization's Project Manager (Plainwell TCRA Project Coordinator):	 Signature Steve Garbaciak, P.E., ARCADIS BBL
Investigative Organization's Project QA Officer:	 Signature Dennis Capria, ARCADIS BBL

QAPP Worksheet #1 Title and Approval Page

Lead Organization's Program Manager for the SRI/FSs:	
	Signature
	James Saric, USEPA Region 5
Lead Organization's Program Manager for the TCRA:	
	Signature
	Sam Borries, USEPA Region 5

Document Control Number: **KRSG-001**

QAPP Worksheet #2 QAPP 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) three miles of Portage Creek and the Kalamazoo River from the city of Kalamazoo to Lake Michigan
Site Number/Code:	MID006007306
Operable Unit:	Including, but not limited to, Operable Unit 5
Contractor Name:	NA
Contractor 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 (2005)
Identify regulatory program:	Comprehensive Environmental Response, Compensation and Liability Act (CERCLA)
Identify approval entity:	USEPA Region 5
Indicate whether the QAPP is a generic or a project-specific QAPP?	This Multi-Area QAPP is a project-specific QAPP to serve the needs of the SRI/FS activities in Area 1, as described in the Area 1 SRI/FS Work Plan (ARCADIS BBL 2007a)and the Plainwell TCRA as described in the TCRA Design Report (ARCADIS BBL 2007b). This QAPP will be updated as required for future work in other areas.
List dates of scoping sessions that were held:	March 19, 2007 at USEPA Region 5 offices in Chicago, IL.
List dates and titles of QAPP documents written for previous site work, if applicable:	

QAPP Worksheet #2 QAPP Identifying Information

Quality Assurance Project Plan	Blasland & Bouck Engineers, P.C. (BBEPC), 1993a, Quality Assurance Project Plan, June 1993. The document was approved by USEPA Region V and Michigan Department of Natural Resources (MDNR).
List organizational partners (stakeholders) and connection with lead organization:	Kalamazoo River Study Group (KRSG), the Respondents under the AOC; and the Michigan Department of Environmental Quality (MDEQ); MDNR, United States Fish and Wildlife Service (USFWS), National Oceanic & Atmospheric Administration (NOAA)
List data users:	The Respondents (Millennium Holdings, LLC and Georgia-Pacific, LLC), USEPA Region 5
Lead Organization's Program Manager:	James Saric, USEPA Region 5, SRI/FS Program Manager, Sam Borries, USEPA Region 5 TCRA Program Manager

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	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 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	- Table of Contents - QAPP Identifying Information	The Table of Contents is provided following the QAPP cover page. Worksheet #2 QAPP Identifying Information
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	Worksheet #3 Distribution List and Worksheet #4 Project Personnel Sign-Off
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	Worksheet #5 Project Organization Chart, Worksheet #6 Communication Pathways, Worksheet #7 Personnel Responsibilities and Qualifications and Worksheet #8 Special Personnel Training Requirements
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History and Background - Site Maps (historical and present)	Worksheet #8 Special Personnel Training Requirements, Worksheet #9 Project Team Planning Sessions Participants Sheet and Worksheet #10-1 through #10-2 Problem Definition for Project DQOs Site history and more detail concerning the project DQOs can be found in the *Supplemental Remedial Investigation/Feasibility Study Work Plan,* February 2007 and *Former Plainwell Impoundment Time-Critical Removal Action Design Report* (TCRA Design Report). February 2007

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
		Site maps can be found in Figure 1-1 Kalamazoo River — Morrow Lake to Lake Michigan and Figure 1-2 Site Location Map (TCRA)
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific Project Quality Objectives (PQOs) - Measurement Performance Criteria Table	Worksheet #11-1 and #11-2 Project Quality Objectives/Systematic Planning Process Statements and Worksheet #12-1 though #12-9 Measurement Performance Criteria for project analytes Details concerning the project objects can be found in the *Supplemental Remedial Investigation/Feasibility Study Work Plan,* February 2007 and *Former Plainwell Impoundment Time-Critical Removal Action Design Report* (TCRA Design Report). February 2007
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	Worksheet #13 Secondary Data Criteria and Limitations Details concerning the secondary data are also discussed in the Administrative Settlement Agreement and Order on Consent (SRI/FS AOC), CERCLA Docket No. V-W-07-C-864. United States Environmental Protection Agency Region 5. Effective February 21, 2007

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table	Worksheet #14 Summary of Project Tasks, QAPP #15-1 through #15-6 Reference Limits and Evaluation for specific monitoring activities and Worksheet #16-1 through #16-2 Project Schedule/Timeline More details concerning the project schedule can be found in the *Master Schedule for the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site*. March 2007

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	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 Containers 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 Sampling Design and Rationale, Worksheet #18-1 through #18-2 Sampling Locations and Methods/SOP Requirements for the project, QAPP Worksheet #19 Analytical SOP Requirements (Sample Containers Preservation and Holding Times), Worksheet #20 Sample Quantities and Control Frequencies, Worksheet #21 Field Sampling SOP References and Worksheet #22 Field Equipment Calibration, Maintenance, Testing and Inspection More details concerning the sampling design and rational can be found in the *Supplemental Remedial Investigation/Feasibility Study Work Plan,* February 2007 and *Former Plainwell Impoundment Time-Critical Removal Action Design Report* (TCRA Design Report). February 2007 The analytical SOPs can be found in Attachment 1-3 More details concerning the filed sampling procedures can be found in the *Multi-Area Field Sampling Plan for the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site* (Multi-Area FSP). April 2007

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
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 analytical SOPs can be found in Attachment 1-3
3.3 Sample Collection Documentation, Handling, Tracking and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	- Sample Collection Documentation Handling, Tracking and Custody SOPs - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody (COC) Form and Seal	Worksheet #27 Sample Custody Requirements More details concerning the field sampling procedures can be found in the *Multi-Area Field Sampling Plan for the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site* (Multi-Area FSP). April 2007 An example of the COC form can be found in Attachment 1-1
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	- QC Samples Table - Screening/Confirmatory Analysis Decision Tree	Worksheet #28-1 through #28-22 present QC sample information for project analytes
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs	Worksheet #29 Project Documents and Records and Worksheet #30 Analytical Services Data Management Plan to be submitted prior to sampling

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
Assessment/Oversight		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table	Worksheet #31 Planned Project Assessments and Worksheet #32 Assessment Findings and Corrective Action Responses Laboratory Certifications can be found in Attachment 1-2
4.2 QA Management Reports	- QA Management Reports Table	Worksheet #33 QA Management Reports
4.3 Final Project Report		

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
Data Review		
5.1 Overview		
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- 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 I) Process, Worksheet #35 Validation (Steps IIa and IIb) Process, Worksheet #36 Validation (Steps IIa and IIb) Summary and Worksheet #37 Usability Assessment
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining	None	NA

QAPP Worksheet #3 Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
James Saric	Project Coordinator	USEPA	312.886.6151	312.353.8426	Saric.James@epa.gov	KRSG-001
Samuel Borries	On-Scene Coordinator	USEPA	312.353.8360	312.353.9306	Borries.Samuel@epa.gov	KRSG-001
Paul Bucholtz	Project Manager	MDEQ	517.373.8174	517.335.4887	BucholtP@michigan.gov	KRSG-001
Sharon Hanshue	Project Coordinator	MDNR	517.335.4058	517.373.0381	HanshuS1@michigan.gov	KRSG-001
Daria Devantier	Chief, Specialized Sampling Unit	MDEQ	517.373.8436	517.335.4887	DevantiD@michigan.gov	KRSG-001
Judith Gapp	Enforcement Specialist	MDEQ	517.373.7402	517.373.2637	GappJ@michigan.gov	KRSG-001
Todd Goeks	Project Coordinator	NOAA	312.886.7527	312.353.5541	Todd.Goeks@noaa.gov	KRSG-001
Lisa Williams, Ph.D.	Project Coordinator	USFWS	517.351.2555	517.351.1443	Lisa_Williams@fws.gov	KRSG-001
Mark Brown, Ph.D.	Technical Contact	Georgia-Pacific Corporation	774.553.5342	NA	mpbrown@gapac.com	KRSG-001

QAPP Worksheet #3 Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Chase Fortenberry, P.G.	Technical Contact	Georgia-Pacific Corporation	404.652.6166	404.654.4701	lcforten@gapac.com	KRSG-001
Mark E. Tapp	Technical Contact — Millennium Holdings, LLC	Lyondell/ Equistar	713.309.7164	713.309.2636	Mark.Tapp@lyondell.com	KRSG-001
Steve Garbaciak, P.E.	TCRA Project Coordinator	ARCADIS BBL	312.332.4937	312.332.4434	Steve.Garbaciak@arcadis-us.com	KRSG-001
Mike Erickson, P.E.	SRI/FS Project Coordinator	ARCADIS BBL	810.229.8594	810.229.8837	Michael.Erickson@arcadis-us.com	KRSG-001
Doug Cowin, P.G.	Mill Property Project Coordinator	ARCADIS BBL	312.332.4937	312.332.4434	Doug.Cowin@arcadis-us.com	KRSG-001
Pat McGuire	Mill Property Project Coordinator	ARCADIS BBL	315.671.9233	315.445.9161	Pat.McGuire@arcadis-us.com	KRSG-001
Matt Bowman	TCRA Field Program Coordinator	ARCADIS BBL	810.229.8594	810.229.8837	Matt.Bowman@arcadis-us.com	KRSG-001

QAPP Worksheet #3 Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Mike Scoville	SRI Field Program Coordinator	ARCADIS BBL	315.671.9387	315.445.9161	Michael.Scoville@arcadis-us.com	KRSG-001
Dennis Capria	Data QA Manager	ARCADIS BBL	315.671.9299	315.446.7485	Dennis.Capria@arcadis-us.com	KRSG-001
John Schell, Ph.D.	HHRA Task Manager	ARCADIS BBL	713.785.1680	713.785.1640	John.Schell@arcadis-us.com	KRSG-001
Ken Jenkins, Ph.D.	Ecological RA Task Manager	ARCADIS BBL	707.776.0865	707.776.0850	Ken.Jenkins@arcadis-us.com	KRSG-001
Richard Price	TCRA Field Program Manager	ARCADIS BBL	315.671.9247	315.671.9450	Richard.Price@arcadis-us.com	KRSG-001
Brian Loomis	SRI Field Program Manager	ARCADIS BBL	810.225.1915	810.229.8837	Brian.Loomis@arcadis-us.com	KRSG-001
Albert Zumbuhl	Database Manager	ARCADIS BBL	315.671.9294	315.446.7485	Albert.Zumbuhl@arcadis-us.com	KRSG-001
Dawn Penniman, P.E.	Assistant Project Manager	ARCADIS BBL	315.671.9229	315.445.3053	Dawn.Penniman@arcadis-us.com	KRSG-001

QAPP Worksheet #3 Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Jim Madison	Laboratory Project Manager	Severn Trent Laboratories	802.660.1990	802.660.1919	jmadison@stl-inc.com	KRSG-001
Kirstin L. McCracken	Laboratory QA Manager	Severn Trent Laboratories	802.660.1990	802.660.1919	kmccracken@stl-inc.com	KRSG-001
Mike Jaeger	Laboratory Project Manager	KAR Laboratories	269.381.9666	269.381.9698	JAEGER@karlabs.com	KRSG-001
Linda Felcyn	Laboratory QA Manager	KAR Laboratories	269.381.9666	269.381.9698	FELCYN@karlabs.com	KRSG-001

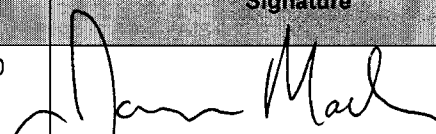
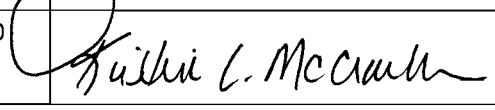
Note:
Copies of the QAPP will be distributed to the individuals above. The copies will consist of the following documents: QAPP, and any subsequent QAPP revisions and addendums.

QAPP Worksheet #4-1 Project Personnel Sign-Off (ARCADIS BBL)

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Mike Erickson, P.E.	SRI/FS Project Coordinator	810.225.1924		6/27/07
Steve Garbaciak, P.E.	TCRA Project Coordinator	312.332.4937		6/27/07
Mike Scoville	SRI Field Program Coordinator	315.671.9387		6/27/07
Brian Loomis	SRI Field Program Manager	810.229.8594		6/27/07
Matt Bowman	TCRA Field Program Coordinator	810.229.8594		6/28/07
Rich Price	TCRA Field Manager	315.671.9247		
Dennis Capria	Data QA Manager	315.671.9299		6/27/07
Albert Zumbuhl	Database Manager	315.671.9294		6/27/07

Note:
The project personnel sign-off table above documents key project personnel who have read the applicable sections of the QAPP and will perform the tasks as described in the QAPP.

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

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Jim Madison	Laboratory Project Manager	802.660.1990		06/27/07
Kirstin L. McCracken	Laboratory QA Manager	802.660.1990		06/27/07

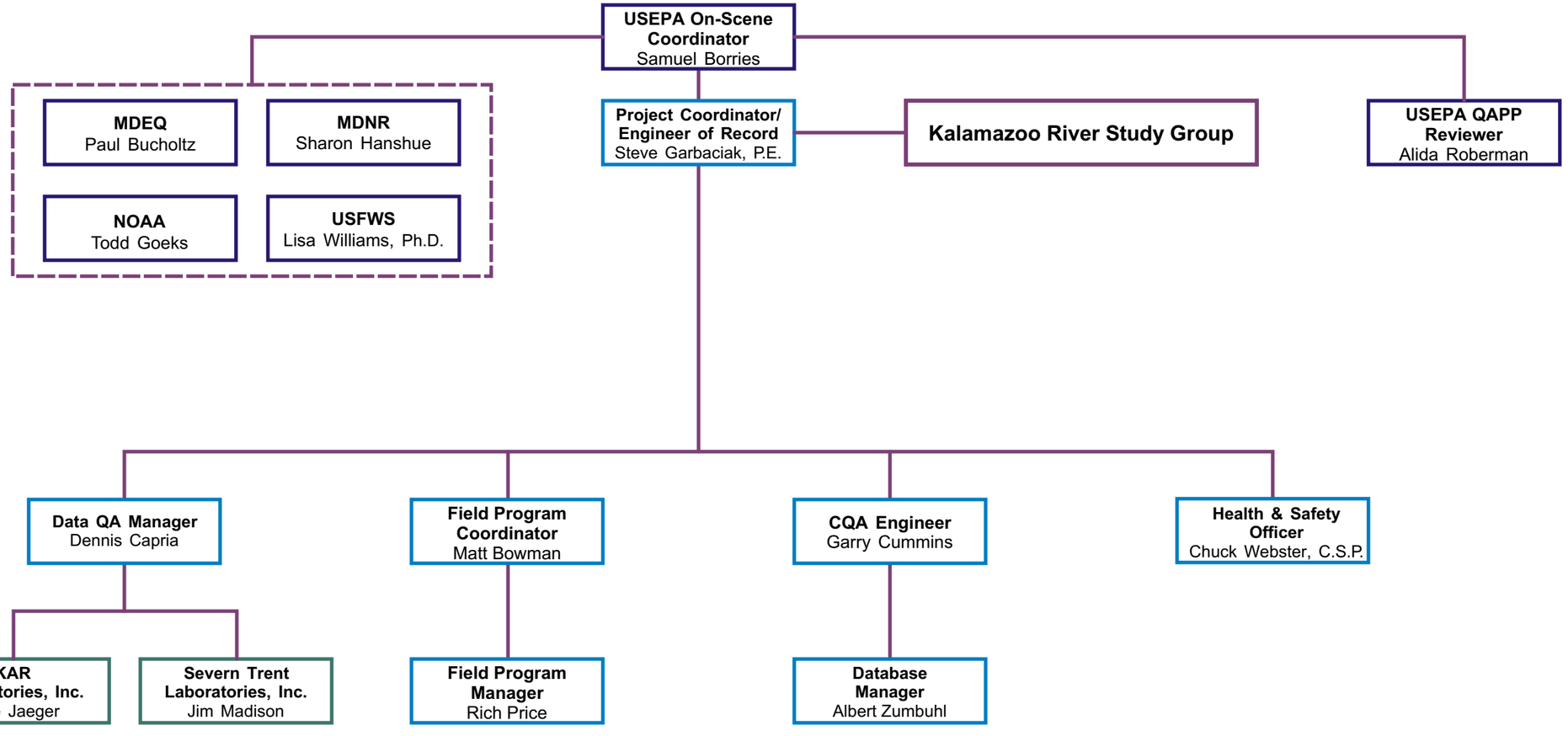
Note:
The project personnel sign-off table above documents key project personnel who have read the applicable sections of the QAPP and will perform required activities in accordance with the QAPP.

QAPP Worksheet #4-3 Project Personnel Sign-Off (KAR)

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Mike Jaeger	Laboratory Project Manager	269.381.9666	Michael Jaeger	8 May 2007
Linda Felcyn	Laboratory QA Manager	269.381.9666	Linda Felcyn	June 15, 2007

Note:
The project personnel sign-off table above documents key project personnel who have read the applicable sections of the QAPP and will perform the tasks as described in the QAPP.

QAPP Worksheet #5-1 Plainwell TCRA Project Organization Chart



KALAMAZOO RIVER STUDY GROUP
ALLIED PAPER, INC./PORTAGE CREEK/
KALAMAZOO RIVER SUPERFUND SITE

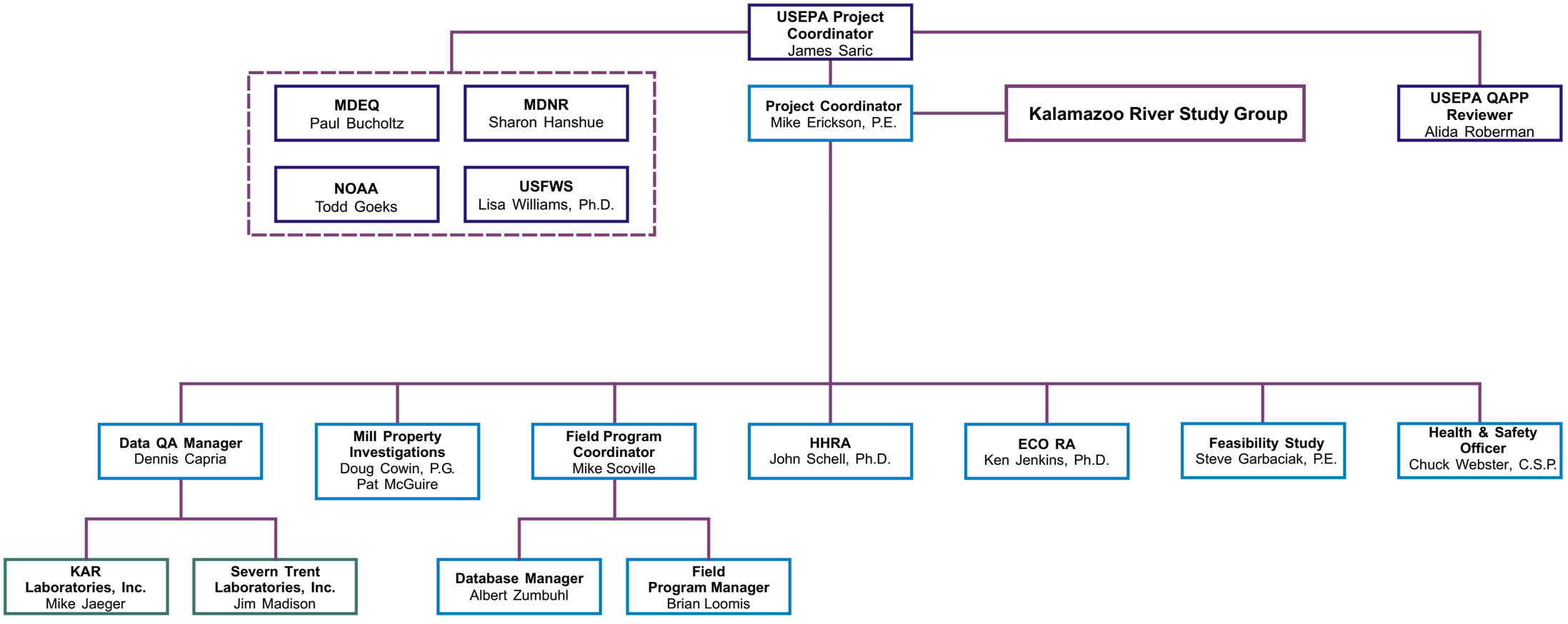
QUALITY ASSURANCE PROJECT PLAN

PLAINWELL TCRA PROJECT ORGANIZATION CHART

ARCADIS BBL
Infrastructure, environment, facilities

06/15/07 SYR-D85-DJH
64530670/64530f02.CDR

QAPP Worksheet #5-2 SRI/FS Project Organization Chart



KALAMAZOO RIVER STUDY GROUP
ALLIED PAPER, INC./PORTAGE CREEK/
KALAMAZOO RIVER SUPERFUND SITE

QUALITY ASSURANCE PROJECT PLAN

SRI/FS PROJECT ORGANIZATION CHART

ARCADIS BBL
Infrastructure, environment, facilities

06/15/07 SYR-85-DJH
64530670/64530f03.CDR

QAPP Worksheet #6 Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (e.g., Timing, Pathways)
Point of Contact with USEPA Remedial Project Manager (RPM)	Respondents' Lead Program Manager	Mike Erickson, P.E.	810.229.8594	Joint TCRA and SRI/FS planning documents will be provided to James Saric, USEPA and Samuel Borries, USEPA by Mike Erickson.
Manage all Project Phases (TCRA)	Project Coordinator	Steve Garbaciak, P.E.	312.332.4434	Steve Garbaciak will be the ARCADIS BBL liaison to all agencies for the Plainwell TCRA.
Manage all Project Phases (SRI/FS)	Project Coordinator	Mike Erickson, P.E.	810.229.8594	Mike Erickson will be the ARCADIS BBL liaison to all agencies for the SRI/FS.
Coordinate SRI/FS Field Program	SRI Field Program Coordinator	Mike Scoville	315.671.9387	To be notified of field-related questions or problems by phone, e-mail, or fax by COB the next business day.
QAPP changes in the field	SRI Field Program Manager	Brian Loomis	810.229.8594	To be notified Mike Scoville by phone and e-mail of any changes to QAPP made in the field and the reasons within 2 business days. Will notify the SRI Field Program Coordinator and the Project Coordinator of any such changes immediately.
Daily Field Progress Reports	SRI Field Program Manager	Brian Loomis	810.229.8594	To be provided daily field progress reports. Will provide complete sets of daily field progress reports, sampling logs, chains-of-custody forms, and other information to the SRI Field Program Coordinator.
Coordinate TCRA Field Program	TCRA Field Program Coordinator	Matt Bowman	810.229.8594	Notify of field-related problems by phone, e-mail, or fax by COB the next business day.

QAPP Worksheet #6 Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (e.g., Timing, Pathways)
QAPP changes in the field	TCRA Field Program Manager	Rich Price	315.671.9247	Notify Matt Bowman by phone and e-mail of changes to QAPP made in the field and the reasons within 2 business days.
Daily Field Progress Reports	TCRA Field Program Manager	Rich Price	315.671.9247	Sampling Team will email or fax directly field progress reports to database manager.
Reporting Lab Data Quality Issues — STL	Laboratory QA Manager	Kirstin McCracken	802.660.1990	All QA/QC issues with project field samples will be reported by Kirstin McCracken to Mike Scoville or Matt Bowman and Dennis Capria within 2 business days.
Reporting Lab Data Quality Issues — KAR	Laboratory QA Manager	Linda Felcyn	269.381.9666	All QA/QC issues with project field samples will be reported by Linda Felcyn to Mike Scoville/Matt Bowman and Dennis Capria within 2 business days.
Field and Analytical Corrective Actions	Data QA Manager	Dennis Capria	315.671.9299	The need for corrective action for field and analytical issues will be determined by Dennis Capria in conjunction with the Project Coordinator, the Field Program Coordinator or the Laboratory QA Manager, as appropriate.
Release of Analytical Data	Data QA Manager	Dennis Capria	315.671.9299	No final analytical data can be released until validation is completed and Dennis Capria has approved the release.
QAPP Amendments	Data QA Manager	Dennis Capria	315.671.9299	Any major changes to the QAPP must be approved by Dennis Capria and the Project Coordinator and USEPA before the changes can be implemented.

QAPP Worksheet #7 Personnel Responsibilities and Qualifications

Name	Title	Organizational Affiliation	Education and Experience Qualifications
James Saric	Project Coordinator	USEPA	Designated as the USEPA's Project Coordinator
Samuel Borries	On-Scene Coordinator	USEPA	Designated as the USEPA's Project Coordinator
Alida Roberman	QAPP Reviewer	USEPA	Designated as the USEPA's Project QA Manager
Steve Garbaciak, P.E.	Plainwell TCRA Project Coordinator	ARCADIS BBL	B.S., Civil Engineering; M.S., Environmental Engineering; 19 years of experience
Mike Erickson, P.E.	SRI/FS Project Coordinator	ARCADIS BBL	B.S.E., Environmental Engineering; M.S.E., Civil Engineering; 11 years of experience
Mike Scoville	SRI Field Program Coordinator	ARCADIS BBL	B.S., Aquatic Environments; minor Biology; M.E.M. Water Resources; 18 years of experience
John Schell, Ph.D.	Human health risk assessment (HHRA) Task Manager	ARCADIS BBL	Ph.D. Toxicology; 22 years of experience
Ken Jenkins, Ph.D.	Ecological risk assessment (ECO RA) Task Manager	ARCADIS BBL	B.S., Biology; Ph.D., Biology; more than 30 years of experience
Dennis Capria	Data QA Manager	ARCADIS BBL	B.S., Biology; minor Chemistry; 19 years of experience

QAPP Worksheet #7 Personnel Responsibilities and Qualifications

Name	Title	Organizational Affiliation	Education and Experience Qualifications
Brian Loomis	SRI Field Program Manager	ARCADIS BBL	B.S., Environmental Science and Earth Science (double major); 12 years of experience
Matt Bowman	TCRA Field Program Coordinator	ARCADIS BBL	B.S., Environmental Engineering, 15 years of experience
Rich Price	TCRA Field Program Manager	ARCADIS BBL	A.A.S., Civil Engineering Technology, 20 years of experience
Albert Zumbuhl	Database Manager	ARCADIS BBL	B.S., Environmental Science; M.S., Natural Resources Management; 10 years of experience
Jim Madison	Laboratory Project Manager	STL	B.S., Geology/Environmental Science; 21 years of experience
Kirstin L. McCracken	Laboratory QA Manager	STL	B.A., Geography; 12 years of experience
Mike Jaeger	Laboratory Project Manager	KAR	B.S., Environmental Science; 26 years of experience
Linda Felcyn	Laboratory QA Manager	KAR	B.S., Chemistry; 26 years of experience

Notes:
The responsibilities of the various team members are summarized below by organization.

ARCADIS BBL

SRI/FS Project Coordinator
Responsibilities and duties include:
- communicate with KRSG, USEPA and other stakeholders
- direct ARCADIS BBL project team
- oversee ARCADIS BBL work products
- provide ARCADIS BBL approval for major project deliverables
- serve as liaison with the ARCADIS BBL TCRA Project Coordinator

Plainwell TCRA Project Coordinator
Responsibilities and duties include:
- monitor and coordinate the project construction activities as defined in the Design Report, with an emphasis on adhering to the objectives of the TCRA
- oversee ARCADIS BBL work products
- perform final review of field data reductions and TCRA activity reports
- verify that corrective actions are taken for deficiencies cited during any audits of site activities
- serve as a liaison with ARCADIS BBL's SRI/FS Project Coordinator

Task Managers
The investigation components will be managed by various Task Managers. Duties of each Task Manager include, as appropriate:
- manage relevant day-to-day activities
- develop, establish and maintain files on relevant site activities
- review data reductions from the relevant site activities
- perform final data review of field data reductions and reports on relevant site activities
- verify that corrective actions are taken for deficiencies cited during audits of relevant site activities
- perform overall QA/QC of the relevant portions of the site activities
- review relevant field records and logs
- instruct personnel working on relevant site activities

Field Program Coordinator
- coordinate field and laboratory schedules pertaining to relevant site activities
- request sample bottles from laboratory
- review field instrumentation, maintenance and calibration to meet quality objectives
- prepare reports pertaining to relevant field activities
- maintain field and laboratory files of notebooks/logs, data reductions and calculations; provide complete copies to the Project Coordinator.

Field Project Manager
Responsibilities and duties include:
- perform field procedures associated with the investigations as set forth in the Work Plan/Design Report
- perform field analyses and collect QA samples
- calibrate, operate and maintain field equipment
- reduce field data
- maintain sample custody
- prepare field records and logs

Data QA Manger
Responsibilities and duties include:
- review laboratory data packages
- oversee and interface with the analytical laboratory
- coordinate field QA/QC procedures with Task Managers (including audits of field activities), concentrating on field analytical measurements and practices to meet data quality objectives (DQOs)
- review field reports
- perform and review audit reports
- prepare interim QA/QC compliance reports
- prepare a QA/QC report in accordance with USEPA guidelines, including an evaluation of field and laboratory data, and data usability reports

Database Manager
Responsibilities and duties include:
- data collected in field will be transcribed from field form or notebooks and tabulated into database
- analytical results provided by the laboratory in the form of an electronic data deliverable (EDD) will imported into the database
- five percent QA/QC check of imported field and laboratory results versus the hardcopy
- tabulation of the data to end users

Analytical Laboratories

General responsibilities and duties of the analytical laboratories include:
- perform sample analyses and associated laboratory QA/QC procedures
- supply sampling containers and shipping cartons
- maintain laboratory custody of sample
- adhere to all protocols in the QAPP

Project Manager
Responsibilities and duties include:
- serve as primary communication link between ARCADIS BBL and laboratory technical staff
- monitor workloads and maintain availability of resources
- oversee preparation of analytical reports
- supervise in-house chain of custody

Laboratory QA Manager
Responsibilities and duties include:
- supervise personnel reviewing and inspecting all project-related laboratory activities
- conduct audits of all laboratory activities

United States Environmental Protection Agency

Project Coordinator/On-Scene Coordinator
Responsibilities and duties include:
- provide the USEPA's reviews and approvals of submitted documents
- coordinate with other agency stakeholders
- monitor progress of site activities

QA Technical Staff
Responsibilities and duties include:
- review and approval of the QAPP
- review of the QA/QC portion of any submitted report
- perform field and laboratory audits, if necessary

QAPP Worksheet #8 Special Personnel Training Requirements

Project Function	Specialized Training	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Field Activities	40-hour HAZWOPER	Certified Training Professionals	NA	Field operations personnel	ARCADIS BBL personnel	ARCADIS BBL project offices
Analytical Chemistry	MDEQ Certification	MDEQ	NA	NA	KAR 4425 Manchester Road Kalamazoo, MI 49001 269.381.9666	KAR 4425 Manchester Road Kalamazoo, MI 49001 269.381.9666
Analytical Chemistry	NELAP Accreditation	Primary Accrediting State	NA	NA	STL Burlington 30 Community Drive, Suite 11 S. Burlington, VT 05403 Jim Madison 802.660.1990	STL Burlington 30 Community Drive, Suite 11 S. Burlington, VT 05403 Jim Madison 802.660.1990
Additional training/certification requirements are listed in the project HSP						

Note:
Current HAZWOPER training certificates will be maintained in a file at the Syracuse office location for each employee performing work at the Site where 40-hour training is required for the position assignment.

QAPP Worksheet #9 Project Team Planning Sessions Participants Sheet

Project Name: **Kalamazoo River Project**	Site Name: **Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site**
Projected Date(s) of Sampling:	Site Location:
Project Manager: Mike Erickson, P.E.	USEPA Region 5 Offices, Chicago, IL
Date of Session: 3/19/07	
Scoping Session Purpose: Review QAPP preparation requirements and approach. Meeting report provided to Shari Kolak on March 19, 2007.	

Name	Title	Affiliation	Phone	E-mail Address	Project Role
Shari Kolak	RPM	USEPA	312.886.6151	Kolak.Shari@epa.gov	Project Coordinator
Alida Roberman	Remedial Project QA/QC Manager	USEPA	312.886.7185	Roberman.Alida@epa.gov	QA Manager
Richard Byvik	QA/QC Manager	USEPA	312.886.7185	Byvik.Richard@ epa.gov	QAPP Reviewer
Paul Bucholtz	Project Manager	MDEQ	517.373.8174	Bucholtp@michigan.gov	Project Manager
Mike Erickson, P.E.	SRI/FS Project Coordinator	ARCADIS BBL	810.229.8594	Michael.Erickson@arcadis-us.com	SRI/FS Project Coordinator
Steve Garbaciak, P.E.	Plainwell TCRA Project Coordinator	ARCADIS BBL	312.332.4937	Steve.Garbaciak@arcadis-us.com	TCRA Project Coordinator
Dennis Capria	Data QA Manager	ARCADIS BBL	315.671.9299	Dennis.Capria@arcadis-us.com	Data QA Manager

QAPP Worksheet #10-1 Problem Definition — DQOs (TCRA)

Step 1: State the Problem:
Under agreements with the United States Environmental Protection Agency (USEPA) a TCRA will be conducted in the former Plainwell Impoundment to address the area of PCB contaminated sediments, river bank soils and floodplain soils defined in the TCRA Design Report (ARCADIS BBL 2007b). The problem to be addressed is the presence of PCBs in Kalamazoo River at levels deemed unacceptable for ecological and human receptors by removal of materials identified in the TCRA Design Report.
Step 2: Identify the Goal of the Study:
The key goals of post-removal action monitoring include: • Confirmation of the removal of PCB-contaminated floodplain soils known from current data to contain PCB levels greater than 50 parts per million (ppm or milligram per kilogram [mg/kg]) PCBs • Confirmation of the removal of PCB-contaminated soil in excess of 4 ppm PCBs from the river's north floodplain on or near residential properties upstream of US-131, to the extent that the floodplain can be reasonably accessed • Establishment of reference drawings showing topography of the river channel post-removal action, and the success of revegetation with native plant species The key goals of monitoring during construction operations include: • Comparing downstream turbidity levels to upstream measurements • Collection of water column PCB data • Collection of wastewater treatment discharge for comparison of PCB and miscellaneous parameter concentrations to allowable levels • Analysis of bulk chemistry and physical properties of backfill materials

QAPP Worksheet #10-1 Problem Definition — DQOs (TCRA)

Step 3: Identify Information Inputs:

Decision inputs incorporate both the concentration and distribution of PCBs in site media. A fundamental basis for decision making is that a sufficient number of data points of acceptable quality must be available from the investigation to support the decision. Thus, the necessary inputs for the decision are: 1) the proportion of nonrejected (usable) data points and 2) the quantity of data needed to evaluate whether removal action requirements are satisfied. Input data will originate from samples or measurements of:

- Post-removal floodplain and bank soils PCB concentrations
- Water column measurements of turbidity and PCBs and miscellaneous parameters
- Survey data

The data will be evaluated for completeness, general conformance with requirements of this QAPP and consistency among data sets and with historical data, as appropriate.

Step 4: Define the Boundaries of the Sampling:

The area that includes the removal action will take place in the former Plainwell Impoundment, which is located in Gun Plain and Otsego Townships, downstream of Plainwell, Michigan. It is roughly bounded on its upstream (or southeastern) end by the Main Street Bridge in Plainwell, and on its downstream (or northwestern) end by the Plainwell Dam (see Figure 1-2 and Drawing G-2.1 included in the TCRA Design Report).

Step 5: Develop the Analytic Approach:

Confirmation sampling analysis will be provided within 24 hours of receipt of samples by the lab. These data will be used to adjust removal activities in the field as appropriate. Final decision on removal completeness will be based on validation results. Following validation, the data will be flagged, as appropriate, and any use restrictions will be noted. The loss of any single data point following validation will not hinder use of the overall data set. A decision rule is adopted that 90 percent of the data points not be rejected or deemed unusable as a condition for use of the data set for decision-making purposes. The usable data will be evaluated versus the performance standards. The required reporting limits are also documented in Worksheets 15 so that the lowest achievable detection limit will be reported by the laboratory.

QAPP Worksheet #10-1 Problem Definition — DQOs (TCRA)

Step 6: Specify Performance or Acceptance Criteria:
Specifications for this step call for: 1) giving forethought to corrective actions to improve data usability and 2) understanding the representative nature of the sampling design. The sampling and analysis program described in the TCRA Design Report (ARCADIS BBL 2007b) was the basis for preparation of this QAPP in regards to TCRA sampling requirements. Corrective actions are described within this document.
Step 7: Develop the Plan for Obtaining Data:
The overall QA objective is to develop and implement procedures for field sampling — Chain-of-Custody (COC), laboratory analysis and reporting — that will provide results to support the evaluation of site data consistent with requirements identified in the TCRA Design Report (ARCADIS BBL 2007b). Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, data reporting, internal QC, audits, preventive maintenance of field equipment and corrective action are described in other sections of this QAPP. The Design Report involves a phased approach to both sampling and analysis. This provides the opportunity to evaluate and focus each data collection step to optimize the overall data collection process. A DQO summary for the sampling investigation efforts is presented in the following subsection. The summary consists of stated DQOs relative to data uses, data types, data quantity, sampling and analytical methods, and data measurement performance criteria.

QAPP Worksheet #10-2 Problem Definition — DQOs (SRI/FS)

Step 1: State the Problem:

The problem to be addressed is the presence of PCBs in Kalamazoo River fish tissue at levels deemed unacceptable for both ecological and human receptors.

While the distribution of PCBs in the reach of the river between Morrow Dam and Plainwell has been documented, additional data are needed to fully characterize PCBs in the study area and determine the size and characteristics of potential PCB hot spots. These potential hot spots are represented by locations with the highest sediment PCB concentrations detected in this section of the river. Additional characterization of selected potential hot spots will allow an evaluation of the extent to which these areas may disproportionately influence PCB exposure concentrations and uptake by fish compared to other areas.

For all reaches within the study area where new data is collected, the new data will be used to characterize and assess associated potential risks. The new data will also be used to refine PCB exposure concentrations, provide PCB mass estimates and evaluate changes in surface sediment PCB concentrations through time.

No PCB data for groundwater in the area of the former Plainwell Impoundment are available. PCB data from wells representative of groundwater flowing to the Kalamazoo River that may be impacted by materials to be left in place following the removal action at the former Plainwell Impoundment are needed to assess potential risks associated with PCBs in groundwater, and potential PCB migration in groundwater to the Kalamazoo River.

The TCRA at the former Plainwell Impoundment is designed to address the riverbank PCB source and restore the river to a free-flowing condition. The design of the action does not call for removal of all sediments in the former Plainwell Impoundment, and those sediments remaining after construction is complete are anticipated to be transported downstream. A portion of those sediments that will erode once free-flowing conditions are restored contain PCBs at relatively low levels. Confirmation samples will be taken in remaining sediments to document residual PCB concentrations. Therefore, an understanding of the impacts of the former Plainwell Impoundment removal action on the distribution of PCBs, both in the Plainwell section and in downstream sections, is important to the overall management of the Site. Additionally, residual risks in the former Plainwell Impoundment floodplains and in-stream sediments after the removal action is complete will need to be assessed for the SRI and FS.

QAPP Worksheet #10-2 Problem Definition — DQOs (SRI/FS)

Step 2: Identify the Goal of the Study:

- Generate information sufficient to complete an SRI that fully determines the nature and extent of the release or threatened release of hazardous substances, pollutants, or contaminants at and from the study area; evaluate potential risks to human and ecological receptors; and complete an FS based on the conditions of sections of Portage Creek (between Alcott St. and the Kalamazoo River) and the Kalamazoo River (between Morrow Lake and the Plainwell Dam) under investigation.
- Understand effects of the removal action in the former Plainwell Impoundment on PCB distribution in both the former impoundment and downstream, and the impact on PCB transport.

Step 3: Identify Information Inputs:

In addition to existing data, the following new data will be collected to meet the goals of the SRI/FS:

- surface sediment PCB and total organic carbon (TOC) data from throughout the study area
- sediment core samples from the urbanized Kalamazoo area
- sediment core samples around potential hot spot areas
- sediment and/or soil samples, as appropriate, in the area adjacent to the Crown Vantage Landfill
- sediment core samples from Portage Creek
- bank and floodplain soil samples from the historically inundated area upstream of the Plainwell No. 2 Dam
- sediment core samples and bank soil samples in the vicinity of the Plainwell No. 2 Dam to the former Plainwell Inc. Mill
- sediment PCB, TOC, Target Compound List/Target Analyte List (TCL/TAL), and Acid Volatile Sulfides/Simultaneously Extracted Metals (AVS/SEM) data from the study area
- surface-water sampling upstream and downstream of the former Plainwell Impoundment
- bathymetric data from the former Plainwell Impoundment, collected periodically during and after implementation of the removal action
- regularly collected surface-water flow, PCB, and total suspended solids (TSS) data collected upstream and downstream of the former Plainwell Impoundment during construction activities
- sediment PCB data in the former Plainwell Impoundment following completion of the removal action
- yearling smallmouth bass tissue PCB data from the Otsego City Impoundment, following completion of the removal action in the former Plainwell Impoundment
- groundwater PCB data from wells representative of groundwater flowing to the Kalamazoo River that may have been impacted by materials left in place in the former Plainwell Impoundment, following completion of the removal action

QAPP Worksheet #10-2 Problem Definition — DQOs (SRI/FS)

Step 4: Define the Boundaries of the Sampling:

The general geographic boundaries of the study area are the Kalamazoo River and its floodplain from Morrow Dam in Comstock to Plainwell Dam, and Portage Creek from Alcott Street to the Kalamazoo River.

Temporally, the limits of the study vary according to the specific objectives defined in Step 2.

Step 5: Develop the Analytic Approach:

The analytic approach to completion of the Work Plan goals will incorporate both statistical hypotheses testing and estimation, or other analytic approaches. The analytic approach includes, but is not limited to:

- comparison of new sediment data to historical data to update trend analyses, test previously calculated trends and determine the statistical significance of trends through time
- calculation of updated PCB mass inventories
- geostatistical evaluation of PCB data collected around potential hot spots to assess the extent of these areas
- statistical comparison of average PCB concentration to assess the statistical significance of changes in concentrations through time and between locations
- calculation of annual PCB and solids loads at several locations in the SRI study area for analysis of spatial trends in transport, and to compare with historical PCB and solids load estimates
- statistical comparison of surface-water PCB and TSS data from upstream and downstream of the former Plainwell Impoundment during construction
- comparison of sequential bathymetric surveys conducted after construction in the former Plainwell Impoundment is complete to evaluate the movement of mid-channel sediment that will be left behind
- comparison of mean PCB concentrations in yearling bass collected from the Otsego City Impoundment pre- and post-construction
- calculation of groundwater PCB mass flux to the Kalamazoo River

The decision on whether data can be used in the exposure evaluation will be based on validation results. Following validation, the data will be flagged, as appropriate, and any use restrictions will be noted. The Design Work Plan has been devised so that the loss of any single data point will not hinder description of the distribution of constituents of concern or the development of a risk assessment. Given this, a decision rule would be that 90 percent of the data points not be rejected or deemed unusable for exposure evaluation purposes is adopted to govern suitability of the data set for decision-

QAPP Worksheet #10-2 Problem Definition — DQOs (SRI/FS)

making. The usable data will be evaluated versus the performance documented in Worksheet #15. The required reporting limits are also documented in these tables so that the lowest achievable detection limit will be reported by the laboratory and, when possible, at or below the guidance values and cleanup target levels. Applicable actions will be evaluated, if needed, based on the results of the exposure evaluation.

Step 6: Specify Performance or Acceptance Criteria:

Based on the potential uses of data in the decision-making process, performance criteria and acceptance criteria are equivalent to those associated with data collected as part of the original RI and 2000 Supplemental Investigations. Data quality assurance/quality control (QA/QC) will be managed as described in the FSP and this QAPP. Performance or acceptance criteria for data applications, such as statistical comparisons or trend analysis, will depend on the data results and variance of the data.

Step 7: Develop the Plan for Obtaining Data:

The overall QA objective is to develop and implement procedures for field sampling — COC, laboratory analysis and reporting — that will provide results to support the evaluation of site data consistent with NCP requirements. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, data reporting, internal QC, audits, preventive maintenance of field equipment and corrective action are described in other sections of this QAPP.

The Work Plan involves a phased approach to both sampling and analysis. This provides the opportunity to evaluate and focus each data collection step to optimize the understanding of PCB distributions at the site afforded by the level of sampling to be conducted.

QAPP Worksheet #11-1 Project Quality Objectives/Systematic Planning Process Statements (TCRA)

Who will use the data?
ARCADIS BBL, their subcontractors and overseeing agencies will use the data to assess the efficacy of the removal action and to confirm adherence to the TCRA performance standards.
What will the data be used for?
The data will be used to confirm that the removal action is performed in accordance with the design and specifications of the TCRA Design Report, to monitor environmental variables during and after construction activities, and to characterize materials generated and used by construction activities.
What type of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)
Data include confirmation sampling for PCBs in sediment and soil samples, characterization of soil for backfill soil and sediment (VOCs, SVOCs, pesticides, and metals) a wide variety of constituents, evaluation of instream turbidity and PCB concentrations and evaluation of water treatment effluent PCB data. Specific sampling and analyses are described in the TCRA Design Report. Standard protocols for sample collection and handling, sample preparation, and analytical methods will be followed. Standard operating procedures are provided in this document and in the Multi-Area FSP.
How "good" do the data need to be in order to support the environmental decision?
The confirmatory data need to aid in determining whether additional excavations are needed during remediation activities, determining whether design specifications are achieved and determining operational logistics and protocols during construction.
How much data are needed? (number of samples for each analytical group, matrix and concentration)
The number of samples and analyses for each media are summarized in Worksheet #20 and described in the TCRA Design Report.

QAPP Worksheet #11-1 Project Quality Objectives/Systematic Planning Process Statements (TCRA)

Where, when, and how should the data be collected/generated?
Soil and sediment sampling activities consist of collecting samples for field and laboratory analysis using manual equipment. These samples will be collected upon conclusion of excavation in a work area. Samples will be sent to a lab for analytical evaluation and data results will be returned to ARCADIS BBL within 24 hours. During remediation activities, surface-water samples will be evaluated analysis and analytical results will be returned to ARCADIS BBL within three weeks. Surface water will be also be collected daily and sometimes hourly from upstream and downstream of construction areas, and evaluated for turbidity using a hand-held meter operated from a boat.
Who will collect and generate the data?
ARCADIS BBL and their subcontractors.
How will the data be reported?
Most data will be evaluated and used in real time, and then compiled and ultimately reported in a Post-Construction Report.
How will the data be archived?
All data will be archived by ARCADIS BBL in their Syracuse, NY, office.

QAPP Worksheet #11-2 Project Quality Objectives/Systematic Planning Process Statements (SRI/FS)

Who will use the data?
ARCADIS BBL, their subcontractors and overseeing agencies will use the data to develop an SRI/FS and support decision-making for the Site.
What will the data be used for?
The data will be used to characterize the nature and extent of PCBs in the Kalamazoo River between Morrow Dam and Plainwell Dam, to support risk assessments, for a feasibility study for the Kalamazoo River between Morrow Dam and Plainwell Dam, and to assess the impacts from construction activities at Plainwell, if any.
What type of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)
Data include physical data (e.g., sediment probing, bank survey), and analytical data for sediment and soil. PCBs are the primary constituent of concern, with select samples analyzed for TCL/TAL. Other analyses include particle-size distribution and TOC. Laboratory analyses will be conducted at an off-site laboratory. In addition to sediment and soil, surface-water samples will be collected near Plainwell and analyzed for TSS and PCBs, and yearling fish will be collected downstream of Plainwell and analyzed for PCBs and lipid content. Specific sampling and analyses are described in the Area 1 SRI/FS Work Plan (ARCADIS BBL 2007a). Standard protocols for sample collection and handling, sample preparation and analytical methods will be followed. Standard operating procedures for field sampling are provided in this document and in the Multi-Area FSP. Groundwater sampling will be conducted quarterly upon completion of the TCRA. Samples will be collected using plastic tubing and a peristaltic pump. Samples will be sent to an off-site laboratory for analysis.
How "good" do the data need to be in order to support the environmental decision?
The data need comply with the QA/QC requirements of this QAPP to be consistent with the large historical RI database and all CERCLA regulations.
How much data are needed? (number of samples for each analytical group, matrix and concentration)
See Worksheet #20

QAPP Worksheet #11-2 Project Quality Objectives/Systematic Planning Process Statements (SRI/FS)

Where, when and how should the data be collected/generated?
Soil and sediment samples will be collected during the summer and fall 2007 and in spring 2008. Samples to monitor the impacts of construction activities at Plainwell will be collected during the construction seasons of 2007 and 2008. Post-removal sampling will be conducted in 2009 and 2010, including groundwater sampling. Sampling locations are described in the Area 1 SRI/FS Work Plan (ARCADIS BBL 2007a), and in the TCRA Design Report (ARCADIS BBL 2007b). Sampling methods and protocols will be in accordance with the SOPs provided in the FSP. Most samples will be collected using manual equipment.
Who will collect and generate the data?
ARCADIS BBL will collect the environmental samples. STL and KAR Labs will perform the laboratory analyses on the samples.
How will the data be reported?
The data will be reported by the laboratory in laboratory data packages, including paper copies and electronic data deliverables (EDDs). Data will be validated and reported to the Agencies upon completion. Ultimately, all data will be reported in an SRI report for the Site.
How will the data be archived?
All data will be added to the Kalamazoo River database that is maintained in Microsoft Access by ARCADIS BBL.

QAPP Worksheet #12-1 Measurement Performance Criteria (PCBs 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 and F-10	K-1, S-9, and S-22	Precision — Overall	Relative Percent Difference (RPD) < 50%	Field duplicate	S&A
		Accuracy/Bias	% Recovery (%R) lab-generated limits*	Surrogate	A
		Accuracy/Bias Contamination	< Reporting limit (RL)	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R lab-generated limit*	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

Notes:
[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
[3]MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD are supplied.
* See Attachment 1-3.

QAPP Worksheet #12-2 Measurement Performance Criteria (PCBs Soil/Sediment/Biota)

Matrix	Soil/Sediment/ Biota
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-4, F-5, F-7, and F-8	K-2,S-6,S-13 and S-24	Precision — Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias	%R, lab-generated limits*	Surrogate	A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R lab-generated limit*	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

Notes:
[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
[3]MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD are supplied.
* See Attachment 1-3.

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

Matrix	Soil/Sediment
Analytical Group	VOCs
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-4 and F-5	K-6, S-1 and S-17	Precision — Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias	%R, lab-generated limits*	Surrogate	A
		Accuracy/Bias Contamination	< RL	Blanks (field, trip, equipment, method)	S&A
		Accuracy/Bias	%R lab-generated limit*	LCS	A
		Accuracy/Bias	% Relative abundance, see analytical SOP	Instrument performance check: bromofluorobenzene (BFB)	A
		Precision	Area response & retention times, see analytical SOP	Internal standard	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

Notes:
[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
[3]MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD are supplied.
*See Attachment 1-3.

QAPP Worksheet #12-4 Measurement Performance Criteria (SVOCs Soil/Sediment)

Matrix	Soil/Sediment
Analytical Group	SVOCs
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-4 and F-5	K-5, S-2, and S-16	Precision — Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias	%R lab-generated limits*	Surrogate	A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R lab-generated limit*	LCS	A
		Accuracy/Bias	% Relative abundance, see analytical SOP	Instrument performance check: decafluorotri-phenylphosphine (DFTPP)	A
		Precision	Area response & retention times, see analytical SOP	Internal standard	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

Notes:
[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
[3]MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD are supplied.
* See Attachment 1-3.

QAPP Worksheet #12-5 Measurement Performance Criteria (Pesticides Soil/Sediment)

Matrix	Soil/Sediment
Analytical Group	Pesticides
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-4 and F-5	S-3 and S-15	Precision — Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias	%R lab-generated limits*	Surrogate	A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A
		Accuracy/Bias	%R lab-generated limit*	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

Notes:
[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
[3]MS and MSD must be client-provided. LCS/LCSD performed when no MS/MSD are supplied.
* See Attachment 1-3.

QAPP Worksheet #12-6 Measurement Performance Criteria (Metals Water)

Matrix	Aqueous
Analytical Group	Metals
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 and F-10	S-4	Precision — Overall	RPD < 50%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, calibration, prep.)	S&A
		Accuracy/Bias	%R (90-110)	Initial and Continuing calibration verification	A
		Precision — lab	Certain metals (%R [80-120]) see analytical SOP	Interference check sample (A and AB)	A
		Accuracy/Bias	%R (75-125)	MS/MSD	A
		Precision	%RPD <20%	MS/MSD	A
		Accuracy/Bias	%R (80-120)	LCS	A
		Accuracy/Bias	%R (75-125)	Post-digestion spike	A
		Precision	% Difference (%D) < 10%	Serial dilution[3]	A

Notes:
[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
[3]Performed as needed only for analytes with concentration > 50 times the MDL.

QAPP Worksheet #12-7 Measurement Performance Criteria (Wet Chemistry, Water)

Matrix	Aqueous
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-1 and F-10	K-3,K-4,K-7, K-8, S-10, S-11, S-12, S-23,	Precision — Overall	RPD < 50%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, calibration, prep.)	S&A
		Accuracy/Bias	%R (90-110)	Initial and continuing calibration verification	A
		Accuracy/Bias	%R (75-125)	MS	A
		Accuracy/Bias	%R (75-125)	MSD	A
		Precision	%RPD <20%	Laboratory Duplicate or MS/MSD	A
		Accuracy/Bias	%R lab-generated limit*	LCS	A

Notes:
[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
* See Attachment 1-3.

QAPP Worksheet #12-8 Measurement Performance Criteria (Wet Chemistry, Soil/Sediment/Biota)

Matrix	Soil/Sediment/ Biota
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-4, F-5, F-7, and F-8	S-7 and S-26	Precision — Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, calibration, prep.)	S&A
		Accuracy/Bias	%R (90-110)	Initial and continuing calibration verification	A
		Accuracy/Bias	%R (75-125)	MS	A
		Accuracy/Bias	%R (75-125)	MSD	A
		Precision	%RPD <35%	Laboratory Duplicate or MS/MSD	A
		Accuracy/Bias	%R lab-generated limit*	LCS	A

Notes:
[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
* See Attachment 1-3.

QAPP Worksheet #12-9 Measurement Performance Criteria (Metals 6000-7000 Soil/Sediment)

Matrix	Soil/Sediment
Analytical Group	Metals
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-4 and F-5	S-4 S-18, S19, and S-27	Precision — Overall	RPD < 100%	Field duplicate	S&A
		Accuracy/Bias Contamination	< RL	Blanks (field, equipment, calibration, prep.)	S&A
		Accuracy/Bias	%R (90-110)	Initial and continuing calibration verification	A
		Precision — lab	Certain metals (%R [80-120] see analytical SOP	Interference check sample (A and AB)	A
		Accuracy/Bias	%R (75-125)	MS	A
		Accuracy/Bias	%R (75-125)	MSD	A
		Precision	%RPD <20	Laboratory Duplicate or MS/MSD	A
		Accuracy/Bias	%R lab-generated limit*	LCS	A
		Accuracy/Bias	%R (75-125)	Post-digestion spike	A
		Precision	%D < 10%	Serial dilution[3]	A

Notes:
[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
[3]Performed as needed only for analytes with concentration > 50 times the MDL.
*See Attachment 1-3.

QAPP Worksheet #13 Secondary Data Criteria and Limitations

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 Probing Data	*Technical Memorandum 10: Sediment Characterization/ Geostatistical Pilot Study* (BBL 1994)	KRSG	Site characterization	USEPA approval[1,2]
Sediment PCB, TOC and Particle Size Data – Frozen Cores	Appendix C of the Phase I RI (BBL 2000)	KRSG	Site characterization	USEPA approval[1,2]
Sediment and Floodplain Soils PCB and TOC Data – Focused Samples	Appendix E of the Phase I RI (BBL 2000)	KRSG	Site characterization	USEPA approval[1,3]
Floodplain Soil Data	*Technical Memorandum 3: Results of the Floodplain Soils Investigation* (BBL 1994) *Addendum to Technical Memorandum 3: Supplemental Floodplain Soils Investigation (Portage Creek)* (BBL 1996)	KRSG	Site characterization	USEPA approval[1,2]
Exposed Sediment PCB and TOC Data	*Technical Memorandum 12: Former Impoundment Sediment and Geochronologic Dating Investigation* (BBL 1994)	KRSG	Site characterization	USEPA approval[1,2]
Mill Soils PCB Data	*Technical Memorandum 15: Mill Investigations* (BBL 1996)	KRSG	Site characterization	USEPA approval[1,2]

QAPP Worksheet #13 Secondary Data Criteria and Limitations

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
Fish Data, Multiple Locations	*Technical Memorandum 14: Biota Investigation* (BBL 1994) *Addendum 3 to Technical Memorandum 14: Biota Investigation* (1997 Fish Data) (BBL 1998) Appendix S-2 of the Supplement to the RI/FS (BBL 2000)	KRSG	Site characterization	USEPA approval[4]
Exposed Sediment Earthworm and Mouse Sampling	*Technical Memorandum 2: Results of Phase I TBSA Soil Sampling* (BBL 1994) *Addendum to Technical Memorandum 2: Results of the Phase 2 TBSA Soil Sampling* (BBL 2000)	KRSG	Site characterization	USEPA approval[4]
OSI Bathymetric data and maps	Letter to Brian von Gunten of MDEQ from Mike Scoville of BBL, April 5, 2001	KRSG	Site characterization	USEPA approval
Sediment Erodibility Maps	Appendix S-4 of the Supplement to the RI/FS (BBL 2000)	KRSG	Site characterization	USEPA approval
Surface Water PCB Data	*Technical Memorandum 16 – Surface Water Investigation* (BBL 1995) Appendix S-11 of the Supplement to the RI/FS (BBL 2000)	KRSG	Site characterization	USEPA approval[1] and 1993/1994 data[2]
Erosion Pin Measurements	*Erosion Pin Monitoring Data: Fall 2000 – Fall 2002* (BBL 2003) *Tech Memo – Results of the March 2001 Survey of Erosion Pins* (BBL 2001)	KRSG	Site characterization	USEPA approval

QAPP Worksheet #13 Secondary Data Criteria and Limitations

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
Ecological Risk Assessment Sampling	Appendix S-9 of the Supplement to the RI/FS (BBL 2000) – *Draft Interim Ecological Risk Assessment of Former Impoundment Soils* (Giesy Ecotoxicology, Inc. 2000)	KRSG	Site characterization	USEPA approval
Finely-sectioned Sediment PCB, TOC and Radionuclide Data	Appendix S-7 of the Supplement to the RI/FS (BBL 2000)	KRSG	Site characterization	USEPA approval[1]
Diver Survey of Lake Allegan	Appendix S-5 of the Supplement to the RI/FS (BBL 2000)	KRSG	Site characterization	USEPA approval
Sediment Transect Probing Data	*Phase II Lower River Investigations (Lake Allegan to Lake Michigan) Database Submittals to Agency* (BBL 2000)	KRSG	Site characterization	USEPA approval[1,5]
Sediment PCB and TOC Data	*Phase II Lower River Investigations (Lake Allegan to Lake Michigan) Database Submittals to Agency* (BBL 2000)	KRSG	Site characterization	USEPA approval[1,5]
Sediment Particle-Size Data	*Phase II Lower River Investigations (Lake Allegan to Lake Michigan) Database Submittals to Agency* (BBL 2000)	KRSG	Site characterization	USEPA approval[1,5]
Floodplain Soil Data	*Phase II Lower River Investigations (Lake Allegan to Lake Michigan) Database Submittals to Agency* (BBL 2000)	KRSG	Site characterization	USEPA approval[1,5]
Kalamazoo Lake Sediment Data	*Phase II Lower River Investigations (Lake Allegan to Lake Michigan) Database Submittals to Agency* (BBL 2000)	KRSG	Site characterization	USEPA approval[1,5]

QAPP Worksheet #13 Secondary Data Criteria and Limitations

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
Pottowattamie Marsh and Ottawa Marsh Soil Data	*Phase II Lower River Investigations (Lake Allegan to Lake Michigan) Database Submittals to Agency* (BBL 2000)	KRSG	Site characterization	USEPA approval[1,5]
Plainwell Bank Profile Survey Data	*Former Plainwell Impoundment Bank Characterization Report* (BBL 2004)	KRSG	Site characterization	USEPA approval[1,5]
Plainwell Top-of-Bank PCB Sampling	*Former Plainwell Impoundment Kalamazoo River* (BBL 2003)	KRSG	Site characterization	USEPA approval[1,5]
Plainwell Habitat Description and Classification	*Former Plainwell Impoundment Bank Characterization Report* (BBL 2004)	KRSG	Site characterization	USEPA approval
Sediment Sampling in Support of Removal Design	*Former Plainwell Impoundment Design documents*	KRSG	Site characterization	USEPA approval[1]
Sediment, Surface Water and Fish Data	*Final Report on the Long-Term Monitoring Program: Results from the 1999 Field Season* (CDM 2001); *Long Term Monitoring Program – Results from the 2000 Field Season* (CDM 2002); *Long Term Monitoring Program – 2001 Field Season Results* (CDM 2002); post-2001 long-term monitoring data from CDM	MDEQ/CDM	Site characterization	USEPA approval
Sediment and Soil Sampling – Plainwell and Otsego City Impoundments	*Removal Assessment Report for Allied Paper-Kalamazoo River Site, Otsego/Plainwell, Michigan* (Weston 2002)	USEPA	Site characterization	None

QAPP Worksheet #13 Secondary Data Criteria and Limitations

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 and Soil Sampling – Plainwell and Otsego City Impoundments	*Removal Assessment Report for Allied Paper-Kalamazoo River Site, Otsego/Plainwell, Michigan* (Weston 2002)	USEPA	Site characterization	None
Flow Data	Online Database Reporting Station Measurements	USGS	Evaluation of river flow and constituent transport	None

Notes:

[1] Collected in accordance with the MDEQ and EPA-approved QAPP (BBEPC, June 1993) and MDEQ-approved FSP (BBEPC, July 1993).
[2] Collected in accordance with the MDEQ-approved Kalamazoo River Work Plan (BBL, July 1993).
[3] Collected at the direction of the MDEQ (April 11, 2000).
[4] Data Collected in accordance with the MDEQ- approved Biota Sampling Plan (CMD, October, 1993) and QAPP (BBEPC, June 1993). 1993 data were collected in accordance with the MDEQ-approved Work Plan (BBL, July 1993).
[5] Data collected at the direction of the MDEQ.

QAPP Worksheet #14 Summary of Project Tasks

Sampling Tasks
TCRA • water resuspension monitoring and control • dredged material dewatering system discharge monitoring • bank and floodplain soil post-removal confirmation sampling • backfill soil sampling • post-construction sediment sampling • global positioning system- (GPS-) based survey of excavation limits and sampling locations • digital photos to document progress • post-construction bathymetric surveys
SRI/FS • soil and sediment sampling using GeoProbe®, barge-based drilling, bridge-based vibracoring, and manual sampling techniques • fish collection (Otsego City Impoundment) using shock boat and standard electrofishing techniques, gill nets, rod and reel, fyke nets and other appropriate methods • groundwater monitoring • GPS-based survey of sampling locations • digital photos of sampling locations and samples recovered, as appropriate

QAPP Worksheet #14 Summary of Project Tasks

Analysis Tasks
TCRA • dewatering discharge and surface water samples for resuspension and monitoring and confirmation samples will be processed, prepared and analyzed by: i. KAR for PCBs, TSS and/or total phosphorus • backfill soil samples will be processed, prepared and analyzed by: i. STL for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), chlorinated pesticides, Resource Conservation and Recovery Act (RCRA) metals and PCBs ii. KAR for TPH • post-construction sediment samples will be processed, prepared and analyzed by: i. STL for PCBs, TOC and particle size
SRI/FS • sediment samples will be processed, prepared and analyzed by: i. STL for PCBs, VOCs , SVOCs, chlorinated pesticides, metals, AVS/SEM metals, TOC and particle size • river bank and floodplain soil samples will be processed, prepared and analyzed by: i. STL for PCBs, TOC and particle size

QAPP Worksheet #14 Summary of Project Tasks

- groundwater samples will be processed, prepared and analyzed by:
    i. STL for PCBs, TOC, total dissolved solids (TDS), TSS and metals (sodium, potassium, magnesium and calcium)
    ii. KAR for chloride, sulfate and alkalinity
- fish samples (Otsego City Impoundment) will be processed, prepared and analyzed by:
    i. STL for PCBs and percent lipids

Quality Control Tasks

The samples will be collected, processed and waste disposed of as documented in field SOPs. The QA samples are described in Worksheet #26.

Secondary Data

See Worksheet #13.

Data Management Tasks

The Data Management Plan (ARCADIS BBL 2007f) describes data management tasks and procedures.

Documentation and Records

Field sample identification — described in the Data Management Plan Field Sampling Plan (FSP) (ARCADIS BBL 2007e).

- **Field documentation** — Field personnel will provide comprehensive documentation covering various aspects of field sampling, field analysis and sample COC. This documentation consists of a record that allows reconstruction of field events and sampling handling to aid in the data review and interpretation process. Documents, records and information relating to the performance of the field work will be retained in the project file.

QAPP Worksheet #14 Summary of Project Tasks

- **Laboratory project files** — The laboratory will establish a file for pertinent data. The file will include correspondence, faxed information, phone logs and COC forms. The laboratory will retain project files and data packages for a period not less than 5 years. KRSG and/or its designated representative will retain copies of the analytical data reports according to the requirements of the AOC.

- **Laboratory logbooks** — Workbooks, bench sheets, instrument logbooks and instrument printouts will be used to trace the history of samples through the analytical process and to document important aspects of the work, including the associated quality controls. As such, logbooks, bench sheets, instrument logs, and instrument printouts will be part of the permanent record of the laboratory. Each page or entry will be dated and initialed by the analyst at the time of entry. Errors in entry will be crossed out in indelible ink with one stroke, corrected without the use of white-out or by obliterating or writing directly over the erroneous entry, and initialed and dated by the individual making the correction. Pages of logbooks that are not used will be completed by lining out unused portions. Information regarding the sample, analytical procedures performed and results of the testing will be recorded on laboratory forms or personal notebook pages by the analyst. These notes will be dated and will also identify the analyst, instrument used and instrument conditions. Laboratory notebooks will be periodically reviewed by the laboratory group leaders for accuracy, completeness and compliance with this QAPP. All entries and calculations will be verified by the laboratory group leader. If all entries on the pages are correct, the laboratory group leader will initial and date the pages. Corrective action will be taken for incorrect entries before the laboratory group leader signs.

- **Computer and hard copy storage** — All electronic files and deliverables will be retained by the laboratory for not less than 5 years; hard copy data packages (or electronic copies) will also be retained for not less than 5 years. KRSG and/or its designated representative will retain copies of the analytical data reports according to the requirements of the AOC.

- **Field data reporting** — Information collected in the field through visual observation, manual measurement and/or field instrumentation will be recorded in field notebooks or data sheets and/or on forms. Such data will be reviewed by the appropriate Field Program Manager for adherence to the Work Plan and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible and (as necessary) incorporated into the data evaluation process. If applicable, field data forms and calculations will be processed and included in appendices to the appropriate reports (when generated). The original field logs documents, and data reductions will be kept in the project file at the ARCADIS BBL office in Brighton, Michigan.

- **Laboratory data reporting** — Data reports for all parameters will include, at a minimum, the following items:

  Narrative: Summary of activities that took place during sample analysis, including the following information:

  - laboratory name and address

  - date of sample receipt

QAPP Worksheet #14 Summary of Project Tasks

- cross reference of laboratory identification number to contractor sample identification
- analytical methods used
- deviations from specified protocol
- corrective actions taken

Included with the narrative will be any sample handling documents, including field and internal chain-of-custody forms, air bills, and shipping tags.

Analytical Results: These will be reported according to analysis type and include the following information, as applicable:

- sample identification (ID)
- laboratory ID
- date of collection
- date of receipt
- date of extraction
- date of analysis
- detection limits

Sample results on the report forms will be corrected for dilutions. Soil and sediment data will be reported on a dry weight basis. Unless otherwise specified, all results will be reported uncorrected for blank contamination.

The data associated with Contract Laboratory Program- (CLP-) equivalent reporting will be expanded to include supporting documentation necessary to provide a CLP-equivalent package. This additional documentation will include, but not be limited to, raw data required to recalculate any result, including instrument printouts and quantitation reports. The report also will include standards used in calibration and calculation of analytical results; sample extraction, digestion, and other preparation logs; standard preparation logs; instrument run logs; and moisture content calculations.

QAPP Worksheet #14 Summary of Project Tasks

- **Data reporting levels are as follows**:

    - **Level 1** — **Minimal Reporting**: Minimal or "results only" reporting is used for analyses that, due either to their nature (i.e., field monitoring) or the intended data use (i.e., preliminary screening), do not generate or require extensive supporting documentation.

    - **Level 2** — **Modified Reporting**: Modified reporting is used for analyses that are performed following standard USEPA-approved methods and QA/QC protocols. Based on the intended data use, modified reporting may require some supporting documentation, but not full CLP or CLP-type reporting.

    - **Level 3** — **Full Reporting**: Full CLP or CLP-type reporting is used for those analyses that, based on the intended data use, require full documentation.

Assessment/Audit Tasks

Performance and systems audits will be completed in the field and laboratory during the site investigations, as described below and in Worksheets #31 and #32.

1. **Field Audits** — The following field performance and systems audits will be completed during this project.

   The appropriate Field Program Manager will monitor field performance. Field performance audit summaries will contain an evaluation of field activities to verify that the activities are performed according to established. Field performance audits may be performed by the ARCADIS BBL Project Coordinator (or his designee). The auditor(s) will review field reports and communicate concerns to the ARCADIS BBL Project Coordinator and/or Field Program Managers, as appropriate.

   The number and frequency of field performance audits conducted will be determined independently by the Project Coordinator or Field Program Manager. The ARCADIS BBL Project Coordinator will conduct field performance audits at a frequency of approximately one per month during field activities. The observations made during field performance audits and any recommended changes/deviations to the field procedures will be recorded and documented.

   In addition, the Data QA Manager will review the rinse and trip blank data to identify potential deficiencies in field sampling and cleaning procedures. In addition, systems audits comparing scheduled QA/QC activities from this QAPP with actual QA/QC activities completed will be performed. The appropriate Field Program Manager and Data QA Manager will periodically confirm that work is being performed consistent with this QAPP and the Work Plan.

QAPP Worksheet #14 Summary of Project Tasks

2. Laboratory Audits

Internal laboratory audits are conducted by the Laboratory QA Manager periodically. As part of the audit, the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the laboratory QA manual and SOPs. Results of the audits are summarized and issued to each department supervisor, Laboratory Manager and Laboratory Director. A systems audit of each laboratory is also performed by the Data QA Manager to determine whether the procedures implemented by each laboratory comply with the QA manual and SOPs.

As a participant in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification, in addition to the laboratory's internal audits. Audits are usually conducted annually and focus on laboratory conformance to the specific program protocols for which the laboratory is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action, if necessary.

ARCADIS BBL will conduct an on-site audit of each laboratory prior to the start of analyses for the project. Additional audits may be performed during the project, as deemed necessary.

3. Corrective Action

Corrective actions are required when field or analytical data are not within the objectives specified in this QAPP. Corrective actions include procedures to promptly investigate, document, evaluate and correct data collection and/or analytical procedures. Field and laboratory corrective action procedures for the actions are described below.

a. Field Procedures

If, during field work, a condition is noted by the field crew that would have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause and corrective action implemented by the Field Program Manager or a designee will be documented on a Corrective Action Form and reported to the appropriate ARCADIS BBL Field Program Manager, QA Manager and Project Coordinator.

Examples of situations that would require corrective actions are provided below:

- protocols as defined by the QAPP and/or Work Plan have not been followed
- equipment is not in proper working order or is not properly calibrated

QAPP Worksheet #14 Summary of Project Tasks

- QC requirements have not been met
- issues resulting from performance or systems audits have not been resolved

Project personnel will continuously monitor ongoing work performance as part of daily responsibilities.

b. Laboratory Procedures

In the laboratory, when a condition is noted to have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause and corrective action taken will be documented and reported to the appropriate Project Manager and Data Quality Manger.

Corrective action may be initiated, at a minimum, under the following conditions:

- protocols as defined by this QAPP have not been followed
- predetermined data acceptance standards are not obtained
- equipment is not in proper working order or calibrated
- sample and test results are not completely traceable
- QC requirements have not been met
- issues resulting from performance or systems audits have not been resolved

Laboratory personnel will continuously monitor ongoing work performance as part of daily responsibilities. Corrective action is initiated at the point where the problem has been identified. At whatever level this occurs (analyst, supervisor, data review, or quality control), it is brought to the attention of the Laboratory QA Manager and, ultimately, the Laboratory Director. Final approval of any action deemed necessary is subject to the approval of the Laboratory Director.

Any corrective action deemed necessary based on system or performance audits, the analytical results of split samples, or the results of data review will be implemented. The corrective action may include sample re-extraction, re-preparation, re-analysis, cleanup, dilution, matrix modification, or other activities.

QAPP Worksheet #14 Summary of Project Tasks

Data Review Tasks
See Worksheets #36 and #37.

QAPP Worksheet #15-1 Reference Limits and Evaluation (Discharge Monitoring Plan)

Analyte	Performance Standard Water[3]	Water (µg/L)	
		Laboratory MDL	Laboratory RL
PCB (608)[1]			
Aroclor-1016 (12674-11-2)	--	0.053	0.10
Aroclor-1221 (11104-28-2)	--	--	0.10
Aroclor-1232 (11141-16-5)	--	--	0.10
Aroclor-1242 (53469-21-9)	--	--	0.10
Aroclor-1248 (12672-29-6)	--	--	0.10
Aroclor-1254 (11097-69-1)	--	--	0.10
Aroclor-1260 (11096-82-5)	--	0.024	0.10
Total PCB (1336-36-3)	0.2 µg/L	--	0.10
Inorganics (Wet Chemistry)[2]			
TSS (USEPA 160.2)	30/45 mg/L	--	1000
Total phosphorus as P (USEPA 365.2)	--	7.0	20

Notes:

1. USEPA. *Appendix A to Part 136 Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater Method 608 — Organochlorine Pesticides and PCBs* . July 1995. CAS No. in parentheses.
2. USEPA. *Methods for Chemical Analysis of Water and Wastes* . EPA/600/4-79/020. EMSL-Cincinnati. 1983.
3. Standards are from 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.

RL = Reporting limit.
MDL = Method detection limit.
µg/L = Micrograms per liter.
mg/L = Milligrams per liter.
PCB = Polychlorinated biphenyls.

QAPP Worksheet #15-2 Reference Limits and Evaluation (Groundwater)

Analyte	Ecological Screening Criteria[4,7] (μg/L)	Reference	Water (µg/L) Laboratory MDL	Water (µg/L) Laboratory RL
PCB (8082)[1,2]				
Aroclor-1016 (12674-11-2)	--	--	0.01	0.05
Aroclor-1221 (11104-28-2)	--	--	--	0.05
Aroclor-1232 (11141-16-5)	--	--	--	0.05
Aroclor-1242 (53469-21-9)	--	--	--	0.05
Aroclor-1248 (12672-29-6)	--	--	--	0.05
Aroclor-1254 (11097-69-1)	--	--	--	0.05
Aroclor-1260 (11096-82-5)	--	--	0.01	0.05
Total PCB (1336-36-3)	0.014/0.00012	5, 6	0.01	0.05
Inorganics 6010[1]				
Calcium (7440-70-2)	NA	--	22	5000
Magnesium (7439-95-4)	NA	--	13	5000
Potassium (7440-09-7)	NA	--	180	5000
Sodium (64742-46-7)	NA	--	530	5000
Inorganics (Wet Chemistry)[1]				
Total Organic Carbon (9060)[1]	NA	--	--	1000
TDS (EPA 160.1)[3]	NA	--	--	5000
TSS (EPA 160.2)[3]	NA	--	--	500
Chloride (EPA 300)[3]	NA	--	44	500
Sulfate (EPA 300)[3]	NA	--	37	1000
Alkalinity (EPA 160.2)[3]	NA	--	--	5000

Notes:

1. USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846 3rd ed.* Washington, DC. 1996.

2. SOP modified by Blasland, Bouck & Lee, Inc. (BBL)/STL consistent with USEPA SW-846 8082 Method. CAS No. in parentheses.

3. USEPA. *Methods for Chemical Analysis of Water and Wastes* . EPA/600/4-79/020. EMSL-Cincinnati. 1983.

4. When the risk-based criterion is less than the target detection limit, the TDL is listed as the criterion. In these cases, two numbers are presented in the cell. The first number is the criterion (TDL) and the second number is the risk-based or solubility value, whichever is lower.

5. USEPA. Office of Water, Office of Science and Technology. *National Recommended Water Quality Criteria.* Washington, DC. 2006.

6. Michigan Department of Environmental Quality. Surface Water Assessment Section. *Rule 57 Water Quality Values.* Wildlife Value. Lansing, Michigan. December 11, 2006.

7. These criteria are for screening purposes only.

RL = Reporting limit.

MDL = Method detection limit.

µg/L = Micrograms per liter.

PCB = Polychlorinated biphenyl.

NA = Criterion value not available or not applicable.

TDS = Total dissolved solids.

TSS = Total suspended solids.

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

Analyte	Ecological Screening Criteria[4, 7] (µg/L)	Reference	Water (µg/L)	
			Laboratory MDL	Laboratory RL
PCB (8082)[1,2]				
Aroclor-1016 (12674-11-2)	--	--	0.01	0.05
Aroclor-1221 (11104-28-2)	--	--	--	0.05
Aroclor-1232 (11141-16-5)	--	--	--	0.05
Aroclor-1242 (53469-21-9)	--	--	--	0.05
Aroclor-1248 (12672-29-6)	--	--	--	0.05
Aroclor-1254 (11097-69-1)	--	--	--	0.05
Aroclor-1260 (11096-82-5)	--	--	0.01	0.05
Total PCB (1336-36-3)	0.014/0.00012	5, 6	0.01	0.05
Inorganics (Wet Chemistry)				
TSS (EPA 160.2)[3]	--	--	--	500

Notes:

1. USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846 3rd ed.* Washington, DC. 1996.
2. SOP modified by BBL/STL consistent with USEPA SW-846 8082 Method. CAS No. in parentheses.
3. USEPA. *Methods for Chemical Analysis of Water and Wastes*. EPA/600/4-79/020. EMSL-Cincinnati. 1983.
4. When the risk-based criterion is less than the target detection limit, the TDL is listed as the criterion. In these cases, two numbers are presented in the cell. The first number is the criterion (TDL) and the second number is the risk-based or solubility value, whichever is lower.
5. USEPA. Office of Water Office of Science and Technology. *National Recommended Water Quality Criteria.* Washington, DC. 2006.
6. Michigan Department of Environmental Quality. Surface Water Assessment Section. *Rule 57 Water Quality Values.* Wildlife Value. Lansing, Michigan. December 11, 2006.
7. These criteria are for screening purposes only.

RL = Reporting limit.
MDL = Method detection limit.
µg/L = Micrograms per liter.
PCB = Polychlorinated biphenyl.
TSS = Total suspended solids.

QAPP Worksheet #15-4 Reference Limits and Evaluation — TCRA (Soil/Sediment)[1]

Analyte	Performance Standard Soil[4] (mg/kg)	Sediment/Soil (mg/kg)		
		Laboratory MDL	Laboratory Low Level RL[1]	Laboratory Med. Level RL[1]
PCB (8082)[2,6]				
Aroclor-1016 (12674-11-2)	--	0.017	0.33	--
Aroclor-1221 (11104-28-2)	--	--	0.33	--
Aroclor-1232 (11141-16-5)	--	--	0.33	--
Aroclor-1242 (53469-21-9)	--	--	0.33	--
Aroclor-1248 (12672-29-6)	--	--	0.33	--
Aroclor-1254 (11097-69-1)	--	--	0.33	--
Aroclor-1260 (11096-82-5)	--	0.017	0.33	--
Total PCB (1336-36-3)	4[5]	--	0.33	--
PCB (8082)[2,3,7]				
Aroclor-1016 (12674-11-2)	--	0.0065	0.05	--
Aroclor-1221 (11104-28-2)	--	--	0.05	--
Aroclor-1232 (11141-16-5)	--	--	0.05	--
Aroclor-1242 (53469-21-9)	--	--	0.05	--
Aroclor-1248 (12672-29-6)	--	--	0.05	--
Aroclor-1254 (11097-69-1)	--	--	0.05	--
Aroclor-1260 (11096-82-5)	--	0.0095	0.05	--
Total PCB (1336-36-3)	4[5]	--	0.05	--
Pesticides (8081)[2,7]				
4,4'-DDD (72-54-8)	95	0.00037	0.0033	--
4,4'-DDE (72-55-9)	45	0.00035	0.0033	--
4,4'-DDT (50-29-3)	57	0.00045	0.0033	--
Aldrin (309-00-2)	1	0.00017	0.0017	--
alpha-BHC (319-84-6) (alpha-Hexachlorocyclohexane)	2.6	0.00016	0.0017	--
alpha-Chlordane (5103-71-9)	31	0.00018	0.0017	--
Chlordane (57-74-9)	31	0.00021	0.0017	
beta-BHC (319-85-7) (beta-Hexachlorocyclohexane)	5.4	0.0024	0.017	--
delta-BHC (319-86-8)	NA	0.00018	0.0017	--
Dieldrin (60-57-1)	1.1	0.00036	0.0033	--
Endosufan sulfate (1031-07-8)	NA	0.00018	0.0017	--
Endosulfan I (959-98-8)	1,400	0.00036	0.0033	--
Endosulfan II (33213-65-9)	1,400	0.00035	0.0033	--
Endrin (72-20-8)	65	0.00046	0.0033	--
Endrin Aldehyde (7421-93-4)	NA	0.00038	0.0033	--
Endrin ketone (53494-70-5)	NA	0.00038	0.0033	--
gamma-BHC (58-89-9) (Lindane)	83	0.00018	0.0017	--
gamma-Chlordane (5103-74-2)	31	0.00018	0.0017	--
Heptachlor (76-44-8)	5.6	0.00024	0.0017	--
Heptachlor epoxide (1024-57-3)	3.1	0.00020	0.0017	--
Methoxychlor (72-43-5)	1,900	0.0021	0.017	--
Toxaphene (8001-35-2)	20	0.0170	0.17	--
Volatile Organic Compounds (8260)[2,7]				
1,1,1-Trichloroethane (71-55-6)	460 (C)	0.00017	0.005	0.50
1,1,2,2-Tetrachloroethane (79-34-5)	53	0.00032	0.005	0.50
1,1,2-Trichloro-1,2,2-trifluoroethane (76-13-1)	550 (C)	0.00026	0.050	0.50
1,1,2-Trichloroethane (79-00-5)	180	0.00017	0.005	0.50
1,1-Dichloroethane (75-34-3)	890 (C)	0.00013	0.005	0.50
1,1-Dichloroethene (75-35-4)	200	0.00024	0.005	0.50
1,2,3-Trichlorobenzene (87-61-6)	NA	0.00027	0.005	0.50
1,2,4-Trichlorobenzene (120-82-1)	990	0.00042	0.005	0.50
1,2-Dibromo-3-chloropropane (96-12-8)	1.2 (C)	0.00037	0.005	0.50
1,2-Dibromoethane (106-93-4) (Ethylene dibromide)	92	0.00015	0.005	0.50
1,2-Dichlorobenzene (95-50-1)	210 (C)	0.00031	0.005	0.50
1,2-Dichloroethane (107-06-2)	91	0.00016	0.005	0.50
1,2-Dichloropropane (78-87-5)	140	0.00024	0.005	0.50
1,3-Dichlorobenzene (541-73-1)	170 (C)	0.00034	0.005	0.50
1,4-Dichlorobenzene (106-46-7)	400	0.00032	0.005	0.50

QAPP Worksheet #15-4 Reference Limits and Evaluation — TCRA (Soil/Sediment)[1]

Analyte	Performance Standard Soil[4] (mg/kg)	Sediment/Soil (mg/kg)		
		Laboratory MDL	Laboratory Low Level RL[1]	Laboratory Med. Level RL[1]
Volatile Organic Compounds (8260)[2,7] Cont'd				
2-Butanone (78-93-3)	27,000 (C)	0.0011	0.005	0.50
2-Hexanone (591-78-6)	2,500 (C)	0.00049	0.005	0.50
4-Methyl-2-pentanone (108-10-1)	27,000 (C)	0.00018	0.005	0.50
Acetone (67-64-1)	23,000	0.00045	0.005	0.50
Benzene (71-43-2)	180	0.00045	0.005	0.50
Bromochloromethane (74-97-5)	NA	0.00016	0.005	0.50
Bromodichloromethane (75-27-4)	110	0.00023	0.005	0.50
Bromoform (75-25-2)	820	0.00016	0.005	0.50
Bromomethane (74-83-9)	320	0.00035	0.005	0.50
Carbon Disulfide (75-15-0)	280 (C)	0.00026	0.005	0.50
Carbon Tetrachloride (56-23-5)	96	0.00024	0.005	0.50
Chlorobenzene (108-90-7)	260 (C)	0.00027	0.005	0.50
Chloroethane (75-00-3)	950 (C)	0.00047	0.005	0.50
Chloroform (67-66-3)	1,200	0.00022	0.005	0.50
Chloromethane (74-87-3)	1,100 (C)	0.00028	0.005	0.50
cis-1,2-Dichloroethene (156-59-2)	640 (C)	0.00017	0.005	0.50
cis-1,3-Dichloropropene (10061-01-5)	10	0.00019	0.005	0.50
Dibromochloromethane (124-48-1)	110	0.00015	0.005	0.50
Dichlorodifluromethane (75-71-8)	1,000 (C)	0.00025	0.005	0.50
Ethylbenzene (100-41-4)	140 (C)	0.00027	0.005	0.50
Isopropylbenzene (98-82-8)	390 (C)	0.00084	0.005	0.50
Methyl tert-Butyl Ether (1634-04-4)	1,500	0.00015	0.005	0.50
Methylene Chloride (75-09-2)	1,300	0.00035	0.005	0.50
Styrene (100-42-5)	400	0.00023	0.005	0.50
Tetrachloroethene (127-18-4)	88 (C)	0.0016	0.005	0.50
Toluene (108-88-3)	250 (C)	0.00053	0.005	0.50
trans-1,2-Dichloroethene (156-60-5)	1,400 (C)	0.00028	0.005	0.50
trans-1,3-Dichloropropene (10061-02-6)	10	0.00021	0.005	0.50
Trichloroethene (79-01-6)	500 (C)	0.00035	0.005	0.50
Trichlorofluoromethane (75-69-4)	560 (C)	0.00023	0.005	0.50
Vinyl Chloride (75-01-4)	38	0.00022	0.005	0.50
Xylenes (Total) (1330-20-7)	150 (C)	--	0.005	0.50
Semivolatile Organic Compounds (8270)[2,7]				
1,1'-Biphenyl (92-52-4)	NA	0.04	0.330	--
1,2,4,5-Tetrachlorobenzene (95-94-3)	77,000	--	0.330	--
2,2'-oxybis(1-Chloropropane) (108-60-1)	NA	0.017	0.330	--
2,4,5-Trichlorophenol (95-95-4)	23,000	0.04	0.830	--
2,4,6-Trichlorophenol (88-06-2)	710	0.046	0.330	--
2,4-Dichlorophenol (120-83-2)	660	0.017	0.330	--
2,4-Dimethylphenol (105-67-9)	11,000	0.041	0.330	--
2,4-Dinitrophenol (51-28-5)	NA	0.053	0.830	--
2,4-Dinitrotoluene (121-14-2)	48	0.0096	0.330	--
2,6-Dinitrotoluene (606-20-2)	NA	0.013	0.330	--
2-Chloronapthalene (91-58-7)	56,000	0.014	0.330	--
2-Chlorophenol (95-57-8)	1,400	0.018	0.330	--
2-Methylnaphthalene (91-57-6)	8,100	0.017	0.330	--
2-Methylphenol (95-48-7)	11,000	0.02	0.330	--
2-Nitroniline (88-74-4)	NA	0.029	0.830	--
2-Nitrophenol (88-75-5)	630	0.02	0.330	--
3,3'-Dichlorobenzidene (91-94-1)	66	0.12	0.330	--
3-Nitroaniline (99-09-2)	NA	0.076	0.830	--
4,6-Dinitro-2-methylphenol (534-52-1) (2-Methyl-4, 6-dinitrophenol)	79	0.067	0.830	--
4-Bromophenyl-phenylether (101-55-3)	NA	0.017	0.330	--

QAPP Worksheet #15-4 Reference Limits and Evaluation — TCRA (Soil/Sediment)[1]

Analyte	Performance Standard Soil[4] (mg/kg)	Sediment/Soil (mg/kg)		
		Laboratory MDL	Laboratory Low Level RL[1]	Laboratory Med. Level RL[1]
Semivolatile Organic Compounds (8270)[2,7] Cont'd				
4-Chloro-3-methyphenol (59-50-7)	4,500	0.017	0.330	--
4-Chloroaniline (106-47-8)	NA	0.076	0.330	--
4-Chlorophenyl-phenylether (7005-72-3)	NA	0.019	0.330	--
4-Methylphenol (106-44-5)	11,000	0.031	0.330	--
4-Nitroaniline (100-01-6)	NA	0.091	0.830	--
4-Nitrophenol (100-02-7)	NA	0.052	0.830	--
Acenaphthene (83-32-9)	41,000	0.017	0.330	--
Acenaphthylene (208-96-8)	1,600	0.016	0.330	--
Acetaphenone (98-86-2)	1,100 (C)	0.04	0.330	--
Anthracene (120-12-7)	230,000	0.019	0.330	--
Atrazine (1912-24-9)	71	0.054	0.330	--
Benzaldehyde (100-52-7)	NA	0.04	0.830	--
Benzo(a)anthracene (56-55-3)	20	0.0094	0.330	--
Benzo(a)pyrene (50-32-8)	2	0.013	0.330	--
Benzo(b)fluoranthene (205-99-2)	20	0.025	0.330	--
Benzo(g,h,i)perylene (191-24-2)	2,500	0.021	0.330	--
Benzo(k)fluoranthene (207-08-9)	200	0.033	0.330	--
bis(2-Chloroethoxy) methane (111-91-1)	ND	0.02	0.330	--
bis(2-Chloroethyl) ether (111-44-4)	13	0.015	0.330	--
bis-(2-Ethylhexyl)phthalate (117-81-7)	2,800	0.011	0.330	--
Butylbenzylphthalate (85-68-7)	310 (C)	0.014	0.330	--
Caprolactam (105-60-2)	53,000	0.061	0.330	--
Carbazole (86-74-8)	530	0.015	0.330	--
Chrysene (218-01-9)	2,000	0.013	0.330	--
Dibenzo(a,h)anthracene (53-70-3)	2	0.06	0.330	--
Dibenzofuran (132-64-9)	24,000	0.015	0.330	--
Diethylphthalate (84-66-2)	740 (C)	0.017	0.330	--
Dimethylphthalate (131-11-3)	790 (C)	0.017	0.330	--
Di-n-butylphthalate (84-74-2)	760 (C)	0.018	0.330	--
Di-n-octylphthalate (117-84-0)	6,900	0.0093	0.330	--
Fluoranthene (206-44-0)	46,000	0.016	0.330	--
Fluorene (86-73-7)	27,000	0.018	0.330	--
Hexachlorobenzene (118-74-1)	8.9	0.017	0.330	--
Hexachlorobutadiene (87-68-3)	100	0.016	0.330	--
Hexachlorocyclopentadiene (77-47-4)	720 (C)	0.018	0.330	--
Hexachloroethane (67-72-1)	230	0.019	0.330	--
Indeno(1,2,3-cd)pyrene (193-39-5)	20	0.016	0.330	--
Isophorone (78-59-1)	2,400 (C)	0.016	0.330	--
Naphthalene (91-20-3)	16,000	0.013	0.330	--
Nitrobenzene (98-95-3)	100	0.021	0.330	--
N-Nitroso-di-n-propylamine (621-64-7)	1.2	0.022	0.330	--
N-Nitrosodiphenylamine (86-30-6)	1,700	0.017	0.330	--
Pentachlorophenol (87-86-5)	90	0.042	0.830	--
Phenathrene (85-01-8)	1,600	0.014	0.330	--
Phenol (108-95-2)	12,000 (C)	0.085	0.330	--
Pyrene (129-00-0)	29,000	0.017	0.330	--
TPH (8270 and 8260)[2,6]				
DRO	NA	0.46	10	--
GRO	NA	1.5	10	--
TPH	NA	0.46	10	--
Inorganics (6010)[2,7]				
Arsenic (7440-38-2)	7,600	0.25	1.00	--
Barium (7440-39-3)	37,000,000	0.092	20.0	--
Cadmium (7440-43-9)	550,000	0.064	0.50	--
Chromium (7440-47-3)	2,500,000	0.22	1.0	--

QAPP Worksheet #15-4 Reference Limits and Evaluation — TCRA (Soil/Sediment)[1]

Analyte	Performance Standard Soil[4] (mg/kg)	Sediment/Soil (mg/kg)		
		Laboratory MDL	Laboratory Low Level RL[1]	Laboratory Med. Level RL[1]
Inorganics (6010)[2,7] Cont'd				
Lead (7439-92-1)	400	0.23	1.00	--
Selenium (7782-49-2)	2,600	0.31	3.5	--
Silver (7440-22-4)	2,500	0.29	1.0	--
Sodium (64742-46-7)	1,000,000 (D)	77.4	500	--
Thallium (7440-28-0)	35	0.59	2.5	--
Vanadium (7440-62-2)	750	5.0	13	--
Zinc (7440-66-6)	170,000	2.0	10	--
Inorganics (7471)[2,7]				
Mercury (7439-97-6)	160	0.010	0.034	--

Notes:

1. The target reporting limits are based on wet weight. Actual reporting limits will vary based on sample weight and moisture content.
2. USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846 3rd ed.* Washington, DC. 1996.
3. Method 8082 SOP for STL refer to Attachment 1-3.
4. MDEQ. Direct Contact Criteria. *Part 201 Generic Cleanup Criteria and Screening Levels* . January 23, 2006.
   These criteria are for screening purposes only.
5. Residential land use category direct contact value.
6. Analysis performed by KAR.
7. Analysis performed by STL.

RL = Reporting limit.
MDL = Method detection limit.
mg/kg = Milligrams per kilogram.
PCB = Polychlorinated biphenyl.
NA = Criterion value not available or not applicable.
ID = Insufficient data to develop criterion.
C = Value presented is a screening level based on the chemical-specific generic soil saturation concentration (C_{sat}), because the calculated risk-based criterion is greater than C_{sat}.
D = Calculated criterion exceeds 100 percent, hence it is reduced to 100 percent or 1.0E9 parts per billion

QAPP Worksheet #15-5 Reference Limits and Evaluation — SRI/FS (Soil/Sediment)[1]

Analyte	Ecological Screening Criteria[8] (mg/kg)	Reference	Human Health Screening Criteria					Sediment/Soil (mg/kg)		
			EPA SSLs (mg/kg)[8,17]	Route of Exposure	Region 9 PRGs (mg/kg)[8,18]	Region 6 MSSLs (mg/kg)[8,19]	Region 3 RBCs (mg/kg)[8,20]	Laboratory MDL	Laboratory Low Level RL[1]	Laboratory Med. Level RL[1]
PCB (8082)[3,7]										
Aroclor-1016 (12674-11-2)	NA	NA	NA	NA	3.9	3.9	5.5	0.0032	0.017	--
Aroclor-1221 (11104-28-2)	0.12	9	NA	NA	NA	0.22	0.32	0.0023	0.017	--
Aroclor-1232 (11141-16-5)	0.60	9	NA	NA	NA	0.22	0.32	0.0023	0.017	--
Aroclor-1242 (53469-21-9)	0.17	9	NA	NA	NA	0.22	0.32	0.0033	0.017	--
Aroclor-1248 (12672-29-6)	1.0	9	NA	NA	NA	0.22	0.32	0.0054	0.017	--
Aroclor-1254 (11097-69-1)	0.81	9	NA	NA	0.22	0.22	0.32	0.0019	0.017	--
Aroclor-1260 (11096-82-5)	4500	9	NA	NA	NA	0.22	0.32	0.0026	0.017	--
Total PCB	0.06/5.6	10, 11	NA	NA	NA	0.22	0.32	0.0054	0.017	--
Pesticides CLP[6]										
4,4'-DDD (72-54-8)	0.00488	10	3	Ingestion & Dermal	2.4	2.4	2.7	0.0003	0.0033	--
4,4'-DDE (72-55-9)	0.00316	10	2	Ingestion & Dermal	1.7	1.7	1.9	0.0003	0.0033	--
4,4'-DDT (50-29-3)	0.00416	10	2	Ingestion & Dermal	1.7	1.7	1.9	0.0003	0.0033	--
Aldrin (309-00-2)	0.002	9	0.04	Ingestion & Dermal	0.029	0.029	0.038	0.0001	0.0017	--
alpha-BHC (319-84-6)	0.12	9	0.1	Ingestion & Dermal	0.090	0.090	0.10	0.0002	0.0017	--
alpha-Chlordane (5103-71-9)	0.00324	10	NA	NA	NA	NA	NA	0.0002	0.0017	--
beta-BHC (319-85-7)	0.12	9	0.4	Ingestion & Dermal	0.32	0.32	0.35	0.0002	0.0017	--
delta-BHC (319-86-8)	0.12	9	NA	NA	NA	NA	NA	0.0002	0.0017	--
Dieldrin (60-57-1)	0.0019	10	0.04	Ingestion & Dermal	0.030	0.030	0.040	0.0003	0.0033	--
Endosufan sulfate (1031-07-8)	0.0055	9	NA	NA	NA	NA	NA	0.0002	0.0017	--
Endosulfan I (959-98-8)	0.0055	9	NA	NA	NA	NA	NA	0.0003	0.0033	--
Endosulfan II (33213-65-9)	0.0055	9	NA	NA	NA	NA	NA	0.0005	0.0033	--
Endrin (72-20-8)	0.00222	10	23	Ingestion & Dermal	18	18	23	0.0003	0.0033	--
Endrin Aldehyde (7421-93-4)	NA	NA	NA	NA	NA	NA	NA	0.0005	0.0033	--
Endrin ketone (53494-70-5)	NA	NA	NA	NA	NA	NA	NA	0.0004	0.0033	--
gamma-BHC (58-89-9)	0.00237	10	0.4	Ingestion & Dermal	0.44	0.44	0.49	0.0001	0.0017	--
gamma-Chlordane (5103-74-2)	0.00324	10	NA	NA	NA	NA	NA	0.0002	0.0017	--
Heptachlor (76-44-8)	0.068	9	0.1	Ingestion & Dermal	0.11	0.11	0.14	0.0001	0.0017	--
Heptachlor epoxide (1024-57-3)	0.005	9	0.07	Ingestion & Dermal	0.053	0.053	0.070	0.0001	0.0017	--
Methoxychlor (72-43-5)	0.019	9	390	Ingestion & Dermal	310	310	390	0.0020	0.017	--
Toxaphene (8001-35-2)	0.028	9	0.6	Ingestion & Dermal	0.44	0.44	0.58	0.0200	0.17	--
Volatile Organic Compounds CLP[6]										
1,1,1-Trichloroethane (71-55-6)	0.03	9	1,200	Inhalation of Volatiles	1,200	1,400	22,000	0.001	0.005	0.25
1,1,2,2-Tetrachloroethane (79-34-5)	0.94	9	0.6	Inhalation of Volatiles	0.41	0.38	3.2	0.001	0.005	0.25
1,1,2-Trichloro-1,2,2-trifluoroethane (76-13-1)	NA	NA	NA	NA	5,600	5,600	230,000	0.001	0.005	0.25
1,1,2-Trichloroethane (79-00-5)	1.2	9	1	Inhalation of Volatiles	0.73	0.84	11	0.00026	0.005	0.25
1,1-Dichloroethane (75-34-3)	0.027	9	1,200	Inhalation of Volatiles	510	850	16,000	0.001	0.005	0.25
1,1-Dichloroethene (75-35-4)	0.031	9	290	Inhalation of Volatiles	120	280	3,900	0.001	0.005	0.25
1,2,3-Trichlorobenzene (87-61-6)	20	12	NA	NA	NA	NA	NA	0.00058	0.005	0.25
1,2,4-Trichlorobenzene (120-82-1)	9.6	9	610	Inhalation of Volatiles	62	140	780	0.00049	0.005	0.25

QAPP Worksheet #15-5 Reference Limits and Evaluation — SRI/FS (Soil/Sediment)[1]

Analyte	Ecological Screening Criteria[8] (mg/kg)	Reference	Human Health Screening Criteria					Sediment/Soil (mg/kg)		
			EPA SSLs (mg/kg)[8,17]	Route of Exposure	Region 9 PRGs (mg/kg)[8,18]	Region 6 MSSLs (mg/kg)[8,19]	Region 3 RBCs (mg/kg)[8,20]	Laboratory MDL	Laboratory Low Level RL[1]	Laboratory Med. Level RL[1]
Volatile Organic Compounds CLP[6] Cont'd										
1,2-Dibromo-3-chloropropane (96-12-8)	NA	NA	NA	NA	0.46	0.0026	0.20	0.00059	0.005	0.25
1,2-Dibromoethane (106-93-4)	NA	NA	NA	NA	0.032	0.028	0.32	0.003	0.005	0.25
1,2-Dichlorobenzene (95-50-1)	0.34	9	600	Inhalation of Volatiles	600	280	7,000	0.001	0.005	0.25
1,2-Dichloroethane (107-06-2)	0.25	9	0.4	Inhalation of Volatiles	0.28	0.35	7.0	0.002	0.005	0.25
1,2-Dichloropropane (78-87-5)	700	12	9	Ingestion & Dermal	0.34	0.35	9.4	0.002	0.005	0.25
1,3-Dichlorobenzene (541-73-1)	1.7	9	NA	NA	530	69	230	0.001	0.005	0.25
1,4-Dichlorobenzene (106-46-7)	0.34	9	20	Ingestion & Dermal	3.4	3.2	27	0.001	0.005	0.25
1,4-Dioxane (123-91-1)	NA	NA	NA	NA	44	44	58	0.05	0.100	5.0
2-Butanone (78-93-3)	0.27	9	NA	NA	22,000	32,000	47,000	0.00088	0.010	0.50
2-Hexanone (591-78-6)	0.022	9	NA	NA	NA	NA	NA	0.0023	0.005	0.50
4-Methyl-2-pentanone (108-10-1)	0.033	9	NA	NA	5,300	5,800	NA	0.002	0.010	0.50
Acetone (67-64-1)	0.0087	9	7,800	Ingestion & Dermal	14,000	14,000	70,000	0.0017	0.010	0.50
Benzene (71-43-2)	0.16	9	0.8	Inhalation of Volatiles	0.64	0.66	12	0.001	0.005	0.25
Bromochloromethane (74-97-5)	NA	NA	NA	NA	NA	NA	NA	0.001	0.005	0.25
Bromodichloromethane (75-27-4)	NA	NA	10	Ingestion & Dermal	0.82	1.0	10	0.001	0.005	0.25
Bromoform (75-25-2)	NA	NA	52	Inhalation of Volatiles	62	62	81	0.002	0.005	0.25
Bromomethane (74-83-9)	NA	NA	9	Inhalation of Volatiles	3.9	3.9	110	0.001	0.005	0.25
Carbon Disulfide (75-15-0)	0.00085	9	720	Inhalation of Volatiles	360	720	7,800	0.001	0.005	0.25
Carbon Tetrachloride (56-23-5)	0.047	9	0.3	Inhalation of Volatiles	0.25	0.24	4.9	0.001	0.005	0.25
Chlorobenzene (108-90-7)	0.41	9	380	Inhalation of Volatiles	150	270	1,600	0.001	0.005	0.25
Chloroethane (75-00-3)	NA	NA	NA	NA	3.0	3.0	220	0.001	0.005	0.25
Chloroform (67-66-3)	0.022	9	780	Ingestion & Dermal	0.22	0.25	780	0.001	0.005	0.25
Chloromethane (74-87-3)	NA	NA	NA	NA	47	1.3	NA	0.001	0.005	0.25
cis-1,2-Dichloroethene (156-59-2)	0.4	9	780	Ingestion & Dermal	43	43	780	0.001	0.005	0.25
cis-1,3-Dichloropropene (10061-01-5)	0.000051	9	NA	NA	NA	NA	NA	0.001	0.005	0.25
Cyclohexane (110-82-7)	NA	NA	NA	NA	140	140	NA	0.001	0.005	0.25
Dibromochloromethane (124-48-1)	NA	NA	8	Ingestion & Dermal	1.1	1.0	7.6	0.001	0.005	0.25
Dichlorodifluromethane (75-71-8)	NA	NA	NA	NA	94	94	16,000	0.001	0.005	0.25
Ethylbenzene (100-41-4)	3.6	9	400	Inhalation of Volatiles	390	230	7,800	0.001	0.005	0.25
Isopropylbenzene (98-82-8)	NA	NA	NA	NA	570	370	7,800	0.0	0.005	0.25
Methyl Acetate (79-20-9)	NA	NA	NA	NA	22,000	22,000	78,000	0.00057	0.005	0.25
Methyl tert-Butyl Ether (1634-04-4)	NA	NA	NA	NA	32	32	160	0.001	0.005	0.25
Methylcyclohexane (108-87-2)	NA	NA	NA	NA	2,600	140	NA	0.001	0.005	0.25
Methylene Chloride (75-09-2)	0.37	9	13	Inhalation of Volatiles	9.1	8.9	85	0.001	0.005	0.25
Styrene (100-42-5)	300	13	1,500	Inhalation of Volatiles	1,700	1,700	16,000	0.001	0.005	0.25
Tetrachloroethene (127-18-4)	0.41	9	1	Ingestion & Dermal	0.48	0.55	1.2	0.001	0.005	0.25
Toluene (108-88-3)	0.05	9	650	Inhalation of Volatiles	520	520	6,300	0.001	0.005	0.25
trans-1,2-Dichloroethene (156-60-5)	0.4	9	1,600	Ingestion & Dermal	69	120	1,600	0.001	0.005	0.25
trans-1,3-Dichloropropene (10061-02-6)	0.000051	9	NA	NA	NA	NA	NA	0.001	0.005	0.25
Trichloroethene (79-01-6)	0.22	9	0.07	Inhalation of Volatiles	0.053	0.043	1.6	0.001	0.005	0.25

QAPP Worksheet #15-5 Reference Limits and Evaluation — SRI/FS (Soil/Sediment)[1]

Analyte	Ecological Screening Criteria[8] (mg/kg)	Reference	Human Health Screening Criteria					Sediment/Soil (mg/kg)		
			EPA SSLs (mg/kg)[8,17]	Route of Exposure	Region 9 PRGs (mg/kg)[8,18]	Region 6 MSSLs (mg/kg)[8,19]	Region 3 RBCs (mg/kg)[8,20]	Laboratory MDL	Laboratory Low Level RL[1]	Laboratory Med. Level RL[1]
Volatile Organic Compounds CLP[6] Cont'd										
Trichlorofluoromethane (75-69-4)	NA	NA	NA	NA	390	390	23,000	0.001	0.005	0.25
Vinyl Chloride (75-01-4)	NA	NA	0.4	Ingestion & Dermal	0.079	0.043	0.090	0.001	0.005	0.25
Xylenes (Total) (1330-20-7)	0.025	9	160,000	Ingestion & Dermal	270	210	16,000	0.001	0.005	0.25
Xylene (m,p) (179601-23-1)	0.025	9	160,000	Ingestion & Dermal	NA	NA	NA	0.001	0.005	0.25
Xylene (o) (95-47-6)	0.025	9	160,000	Ingestion & Dermal	NA	280	NA	0.001	0.005	0.25
Semivolatile Organic Compounds CLP[6]										
1,1'-Biphenyl (92-52-4)	NA	NA	NA	NA	3,000	3,000	3,900	0.083	0.17	--
1,2,4,5-Tetrachlorobenzene (95-94-3)	NA	NA	NA	NA	18	18	23	0.030	0.17	--
2,2'-oxybis(1-Chloropropane) (108-60-1)	NA	NA	NA	NA	3	2.9	2.9	9.100	0.17	--
2,3,4,6-Tetrachlorophenol (58-90-2)	NA	NA	NA	NA	1,800	1,800	2,300	0.083	0.17	--
2,4,5-Trichlorophenol (95-95-4)	4	13	6,100	Ingestion & Dermal	6,100	6,100	7,800	0.032	0.17	--
2,4,6-Trichlorophenol (88-06-2)	10	12	44	Ingestion & Dermal	6.1	44	58	0.032	0.17	--
2,4-Dichlorophenol (120-83-2)	NA	NA	180	Ingestion & Dermal	180	180	230	0.026	0.17	--
2,4-Dimethylphenol (105-67-9)	NA	NA	1,200	Ingestion & Dermal	1,200	1,200	1,600	0.044	0.17	--
2,4-Dinitrophenol (51-28-5)	20	13	120	Ingestion & Dermal	120	120	160	0.024	0.17	--
2,4-Dinitrotoluene (121-14-2)	NA	NA	0.7	Ingestion & Dermal	120	120	160	0.022	0.17	--
2,6-Dinitrotoluene (606-20-2)	NA	NA	0.7	Ingestion & Dermal	61	61	78	0.031	0.17	--
2-Chloronapthalene (91-58-7)	NA	NA	NA	NA	4,900	3,900	6,300	0.083	0.17	--
2-Chlorophenol (95-57-8)	NA	NA	310	Ingestion & Dermal	63	64	390	0.021	0.17	--
2-Methylnaphthalene (91-57-6)	0.13	9	NA	NA	NA	NA	310	0.083	0.17	--
2-Methylphenol (95-48-7)	0.012	9	3,100	Ingestion & Dermal	3,100	3,100	3,900	0.022	0.17	--
2-Nitroniline (88-74-4)	NA	NA	NA	NA	180	180	NA	0.083	0.33	--
2-Nitrophenol (88-75-5)	NA	NA	NA	NA	NA	NA	NA	0.024	0.17	--
3,3'-Dichlorobenzidene (91-94-1)	NA	NA	1	Ingestion & Dermal	1.1	1.1	1.4	0.083	0.17	--
3-Nitroaniline (99-09-2)	NA	NA	NA	NA	18	NA	NA	0.024	0.33	--
4,6-Dinitro-2-methylphenol (534-52-1)	NA	NA	NA	NA	6.1	NA	NA	0.045	0.33	--
4-Bromophenyl-phenylether (101-55-3)	NA	NA	NA	NA	NA	NA	NA	0.022	0.17	--
4-Chloro-3-methyphenol (59-50-7)	NA	NA	NA	NA	NA	NA	NA	0.029	0.17	--
4-Chloroaniline (106-47-8)	NA	NA	240	Ingestion & Dermal	240	240	310	0.030	0.17	--
4-Chlorophenyl-phenylether (7005-72-3)	NA	NA	NA	NA	NA	NA	NA	0.024	0.17	--
4-Methylphenol (106-44-5)	NA	NA	NA	NA	310	310	390	0.026	0.17	--
4-Nitroaniline (100-01-6)	NA	NA	NA	NA	23	NA	NA	0.083	0.33	--
4-Nitrophenol (100-02-7)	7	12	NA	NA	NA	490	NA	0.038	0.33	--
Acenaphthene (83-32-9)	1.3	9	3,400	Ingestion & Dermal	3,700	3,700	4,700	0.025	0.17	--
Acenaphthylene (208-96-8)	0.16	14	NA	NA	NA	NA	NA	0.023	0.17	--
Acetaphenone (98-86-2)	NA	NA	NA	NA	NA	1,700	7,800	0.083	0.17	--
Anthracene (120-12-7)	0.057	10	17,000	Ingestion & Dermal	22,000	22,000	23,000	0.083	0.17	--
Atrazine (1912-24-9)	NA	NA	NA	NA	2.2	2.2	2.9	0.025	0.17	--
Benzaldehyde (100-52-7)	NA	NA	NA	NA	6,100	6,100	7,800	0.077	0.17	--
Benzo(a)anthracene (56-55-3)	0.108	10	0.6	Ingestion & Dermal	0.62	0.15	0.22	0.083	0.17	--

QAPP Worksheet #15-5 Reference Limits and Evaluation — SRI/FS (Soil/Sediment)[1]

Analyte	Ecological Screening Criteria[8] (mg/kg)	Reference	Human Health Screening Criteria					Sediment/Soil (mg/kg)		
			EPA SSLs (mg/kg)[8,17]	Route of Exposure	Region 9 PRGs (mg/kg)[8,18]	Region 6 MSSLs (mg/kg)[8,19]	Region 3 RBCs (mg/kg)[8,20]	Laboratory MDL	Laboratory Low Level RL[1]	Laboratory Med. Level RL[1]
Semivolatile Organic Compounds CLP[6] Cont'd										
Benzo(a)pyrene (50-32-8)	0.15	10	0.06	Ingestion & Dermal	0.062	0.015	0.022	0.029	0.17	--
Benzo(b)fluoranthene (205-99-2)	0.0272	9	0.6	Ingestion & Dermal	0.62	0.15	0.22	0.040	0.17	--
Benzo(g,h,i)perylene (191-24-2)	0.17	9	NA	NA	NA	NA	NA	0.028	0.17	--
Benzo(k)fluoranthene (207-08-9)	NA	NA	6	Ingestion & Dermal	6.2	1.5	2.2	0.083	0.17	--
bis(2-Chloroethoxy) methane (111-91-1)	NA	NA	NA	NA	NA	NA	NA	0.024	0.17	--
bis(2-Chloroethyl) ether (111-44-4)	NA	NA	0.2	Inhalation of Volatiles	0.22	0.21	0.58	0.029	0.17	--
bis-(2-Ethylhexyl)phthalate (117-81-7)	NA	NA	35	Ingestion & Dermal	35	35	46	0.083	0.17	--
Butylbenzylphthalate (85-68-7)	NA	NA	12,000	Ingestion & Dermal	12,000	240	16,000	0.083	0.17	--
Caprolactam (105-60-2)	NA	NA	NA	NA	31,000	31,000	39,000	0.029	0.17	--
Carbazole (86-74-8)	NA	NA	24	Ingestion & Dermal	24	24	32	0.023	0.17	--
Chrysene (218-01-9)	0.16	10	62	Ingestion & Dermal	62	15	22	0.027	0.17	--
Dibenzo(a,h)anthracene (53-70-3)	0.033	10	0.06	Ingestion & Dermal	0.062	0.015	0.022	0.030	0.17	--
Dibenzofuran (132-64-9)	14	15	NA	NA	150	150	NA	0.023	0.17	--
Diethylphthalate (84-66-2)	100	13	49,000	Ingestion & Dermal	49,000	49,000	63,000	0.026	0.17	--
Dimethylphthalate (131-11-3)	200	12	NA	NA	100,000	100,000	NA	0.083	0.17	--
Di-n-butylphthalate (84-74-2)	200	13	6,100	Ingestion & Dermal	6,100	6,100	7,800	0.083	0.17	--
Di-n-octylphthalate (117-84-0)	NA	NA	1,200	Ingestion & Dermal	2,400	NA	NA	0.022	0.17	--
Fluoranthene (206-44-0)	0.423	10	2,300	Ingestion & Dermal	2,300	2,300	3,100	0.023	0.17	--
Fluorene (86-73-7)	0.077	10	2,300	Ingestion & Dermal	2,700	2,600	3,100	0.022	0.17	--
Hexachlorobenzene (118-74-1)	NA	NA	0.3	Ingestion & Dermal	0.30	0.30	0.40	0.083	0.17	--
Hexachlorobutadiene (87-68-3)	NA	NA	6	Ingestion & Dermal	6.2	6.2	8.2	0.021	0.17	--
Hexachlorocyclopentadiene (77-47-4)	10	13	29	Inhalation of Volatiles	370	370	470	0.083	0.17	--
Hexachloroethane (67-72-1)	NA	NA	35	Ingestion & Dermal	35	35	46	0.028	0.17	--
Indeno(1,2,3-cd)pyrene (193-39-5)	0.078	9	0.6	Ingestion & Dermal	0.62	0.15	0.22	0.025	0.17	--
Isophorone (78-59-1)	NA	NA	510	Ingestion & Dermal	510	510	670	0.029	0.17	--
Naphthalene (91-20-3)	0.176	10	170	Inhalation of Volatiles	56	120	1,600	0.022	0.17	--
Nitrobenzene (98-95-3)	40	12	31	Ingestion & Dermal	20	20	39	0.024	0.17	--
N-Nitroso-di-n-propylamine (621-64-7)	NA	NA	0.07	Ingestion & Dermal	0.069	0.069	0.091	0.030	0.17	--
N-Nitrosodiphenylamine (86-30-6)	20	12	99	Ingestion & Dermal	99	99	130	0.083	0.17	--
Pentachlorophenol (87-86-5)	3	13	3	Ingestion & Dermal	3.0	3.0	5.3	0.022	0.33	--
Phenathrene (85-01-8)	0.204	10	NA	NA	NA	NA	NA	0.023	0.17	--
Phenol (108-95-2)	30	12	18,000	Ingestion & Dermal	18,000	18,000	23,000	0.023	0.17	--
Pyrene (129-00-0)	0.195	10	1,700	Ingestion & Dermal	2,300	2,300	2,300	0.083	0.17	--
Inorganics CLP[6A]										
Aluminum (7429-90-5)	58,000	9	NA	NA	76,000	76,000	NA	1.6	20.0	--
Antimony (7440-36-0)	0.27	16	31	Ingestion & Dermal	31	31	31	0.37	6.0	--
Arsenic (7440-38-2)	9.79	10	0.4	Ingestion & Dermal	0.39	0.39	0.43	0.30	1.0	--
Barium (7440-39-3)	330	16	5,500	Ingestion & Dermal	5,400	16,000	16,000	0.14	20.0	--
Beryllium (7440-41-7)	21	16	160	Ingestion & Dermal	150	150	160	0.025	0.50	--
Cadmium (7440-43-9)	0.36	16	70	Ingestion & Dermal	37	39	39	0.045	0.50	--

QAPP Worksheet #15-5 Reference Limits and Evaluation — SRI/FS (Soil/Sediment)[1]

Analyte	Ecological Screening Criteria[8] (mg/kg)	Reference	Human Health Screening Criteria					Sediment/Soil (mg/kg)		
			EPA SSLs (mg/kg)[8,17]	Route of Exposure	Region 9 PRGs (mg/kg)[8,18]	Region 6 MSSLs (mg/kg)[8,19]	Region 3 RBCs (mg/kg)[8,20]	Laboratory MDL	Laboratory Low Level RL[1]	Laboratory Med. Level RL[1]
Inorganics CLP[6A] Cont'd										
Calcium (7440-70-2)	NA	NA	NA	NA	NA	NA	NA	2.8	500	--
Chromium (7440-47-3)	26	16	230	Ingestion & Dermal	300	210	NA	0.14	1.0	--
Cobalt (7440-48-4)	13	16	NA	NA	900	900	NA	0.19	5.0	--
Copper (7440-50-8)	31.6	10	NA	NA	3,100	2,900	3,100	0.22	2.5	--
Iron (7439-89-6)	NA	NA	NA	NA	23,000	55,000	23,000	4.4	10.0	--
Lead (7439-92-1)	11	16	NA	NA	400	400	NA	0.24	1.0	--
Magnesium (7439-95-4)	NA	NA	NA	NA	NA	NA	NA	3.2	500	--
Manganese (7439-96-5)	460	9	NA	NA	1,800	3,200	1,600	0.032	1.5	--
Nickel (7440-02-0)	22.7	10	1,600	Ingestion & Dermal	1,600	1,600	1,600	0.10	4.0	--
Potassium (7440-09-7)	NA	NA	NA	NA	NA	NA	NA	41	500	--
Selenium (7782-49-2)	1	13	390	Ingestion & Dermal	390	390	390	0.28	3.5	--
Silver (7440-22-4)	2	13	390	Ingestion & Dermal	390	390	390	0.16	1.0	--
Sodium (64742-46-7)	NA	NA	NA	NA	NA	NA	NA	49	500	--
Thallium (7440-28-0)	1	13	6	Ingestion & Dermal	5.2	NA	5.5	0.46	2.5	--
Vanadium (7440-62-2)	7.8	16	550	Ingestion & Dermal	78	390	78	0.20	5.0	--
Zinc (7440-66-6)	50	13	23,000	Ingestion & Dermal	23,000	23,000	23,000	0.40	6.0	--
Inorganics CLP[6A]										
Mercury (7439-97-6)	0.1	12	10	Inhalation of Volatiles	23	23	NA	0.011	0.05	--
Wet Chemistry										
TOC (Lloyd Kahn)[5]	NA	NA	NA	NA	NA	NA	NA	--	500	--
Particle size[2]	NA	NA	NA	NA	NA	NA	NA	--	--	--

QAPP Worksheet #15-5 Reference Limits and Evaluation — SRI/FS (Soil/Sediment)[1]

Notes:

1. The target reporting limits are based on wet weight. Actual reporting limits will vary based on sample weight and moisture content.
2. ASTM.
3. USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846 3rd ed.* Washington, DC. 1996.
4. USEPA. *Methods for Chemical Analysis of Water and Wastes*. EPA/600/4-79/020. EMSL-Cincinnati. 1983.
5. USEPA. *Determination of Total Organic Carbon in Sediment (Lloyd Kahn Method).* July 27, 1988.
6. USEPA. *USEPA Contract Laboratory Program Statement of Work for Organics Analysis Multi-Media, Multi-Concentration SOM01.1. May 2005.*
6A. USEPA. *USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis Multi-Media, Multi-Concentration ILM05.3. March 2004.*
7. Method 8082 Standard Operating Procedure (SOP) for Severn Trent Laboratories, refer to Attachment 1-3.
8. These criteria are for screening purposes only.
9. Jones, D.S., G.W. Suter II, and R.N. Hull. 1997. *Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-Associated Biota: 1997 Revision.* U.S. Department of Energy. ES/ER/TM-95/R4.
10. MacDonald, D.D., C.G., Ingersoll, and T.A. Berger. 2000. *Development and Evaluations of Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems.* Archives of Environmental Contamination and Toxicology 39:20-31.
11. Michigan Department of Environmental Quality. 2003. *Final (Revised) Baseline Ecological Risk Assessment, Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site.*
12. Efroymson, R.A., M.E. Will, and G.W. Suter II. 1997a. *Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Processes: 1997 Revision.* Oak Ridge National Laboratory, Oak Ridge.
13. Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997b. *Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision.* Oak Ridge National Laboratory, Oak Ridge, TN. ES/ER/TM-8.
14. Buchman, M.F. 1999. *NOAA Screening Quick Reference Tables: NOAA HAZMAT Report 99-1.* Coastal Protection and Restoration Division, National Oceanic and Atmospheric Administration. Seattle, Washington.
15. Sverdrup, L.E., A.E. Kelley, P.H. Kroh, T. Nielsen, J. Jensen, J.J. Scott-Fordsmand, J. Stenersen. 2001. E*ffects of Eight Polycyclic Aromatic Compounds on the Survival and Reproduction of the Springtail Folsomia Fimetaria L. (collembola, Isotomidae).*
16. USEPA. 2005. *Ecological Soil Screening Levels, Interim Final*. Office of Solid Waste and Emergency Response. Update of November 2003 document.
17. USEPA. 2006. *Supplemental Guidance for Developing Soil Screening Levels for Superfund Sites, Appendix A; Generic SSLs for Residential Scenarios*.
18. USEPA Region 9. 2004. *Preliminary Remedial Goals*. Residential Soil - Direct Contact Exposure Pathway.
19. USEPA Region 6. 2007. *Human Health Medium-Specific Screening Levels*. Residential Soil Screening Levels.
20. USEPA Region 3. 2006. *Risk-Based Concentrations*. Residential Soil Concentrations.

RL = Reporting limit.
MDL = Method detection limit.
mg/kg = Milligrams per kilogram.
PCB = Polychlorinated biphenyl.
NA = Criterion value not available or not applicable.

QAPP Worksheet #15-6 Reference Limits and Evaluation — SRI/FS (Biota)

Analyte	Ecological Screening Criteria[4,5] (mg/kg)	Human Health Screening Criteria			Biota (mg/kg)	
		Region 9 PRGs (mg/kg)[4,6]	Region 6 MSSLs (mg/kg)[4,7]	Region 3 RBCs (mg/kg)[4,8]	Laboratory MDL	Laboratory RL[1]
PCB (8082)[2,3]						
Aroclor-1016 (12674-11-2)	NA	NA	NA	0.045	0.00551	0.05
Aroclor-1221 (11104-28-2)	NA	NA	NA	0.0016	0.0058	0.05
Aroclor-1232 (11141-16-5)	NA	NA	NA	0.0016	0.00667	0.05
Aroclor-1242 (53469-21-9)	NA	NA	NA	0.0016	0.00667	0.05
Aroclor-1248 (12672-29-6)	NA	NA	NA	0.0016	0.00754	0.05
Aroclor-1254 (11097-69-1)	NA	NA	NA	0.0016	0.00783	0.05
Aroclor-1260 (11096-82-5)	NA	NA	NA	0.0016	0.00928	0.05
Total PCB	1	NA	NA	0.0016	0.00928	0.05
Wet Chemistry						
Lipids	NA	NA	NA	NA	--	--

Notes:

1. The target reporting limits are based on wet weight.
2. USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846 3rd ed.* Washington, DC. 1996.
3. Method 8082 SOP for STL, refer to Attachment 1-3.
4. These criteria are for screening purposes only.
5. Halbrook, R.S., R.J. Aulerich, S.J. Bursian, and L. Lewis, L. 1999. *Ecological Risk Assessment in a Large River-Reservoir: 8. Experimental Study of the Effects of Polychlorinated Biphenyls on Reproductive Success in Mink*. Environ. Toxicol. Chem. 18:649-654.
6. USEPA Region 9. 2004. *Preliminary Remedial Goals*.
7. USEPA Region 6. 2007. *Human Health Medium-Specific Screening Levels*.
8. USEPA Region 3. 2006. *Risk-Based Concentrations*. Region 3 RBCs are for concentrations in edible fish tissue.

RL = Reporting limit.
MDL = Method detection limit.
mg/kg = Milligram per kilogram.
PCB = Polychlorinated biphenyl.

QAPP Worksheet #16-1 TCRA Project Schedule/Timeline

Activities	Organization	Anticipated Date(s) of Initiation	Anticipated Date of Completion	Deliverable	Deliverable Due Date
TCRA					
QAPP, HSP	ARCADIS BBL	February 21, 2007	NA	Yes	April 9, 2007 — QAPP and HSP submitted with respective SRI/FS Multi-Area documents
FSP	ARCADIS BBL	February 21, 2007	NA	Yes	NA — Submitted with SRI/FS Multi-Area FSP
Mobilization (Site preparation activities)	ARCADIS BBL	April 16, 2007	April 20, 2007	No	NA
Removal Area 1 thru Removal Area 13 (Confirmation sampling)	ARCADIS BBL	June 2007	October 2008	No	NA
Closure activities	ARCADIS BBL	October 27, 2008	December 26, 2008	No	NA
Annual reports	ARCADIS BBL	January 1, 2009	NA	Yes	December 31, 2009

QAPP Worksheet #16-2 SRI/FS Project Schedule/Timeline

Activities	Organization	Anticipated Date(s) of Initiation	Anticipated Date of Completion	Deliverable	Deliverable Due Date[1]
Morrow Dam to Plainwell Dam SRI/FS					
Phase 1 field work	ARCADIS BBL	May 2007	June 2007	No	NA
Data processing and agency meetings	ARCADIS BBL	June 2007	August 2007 (approximate)	No	NA
Phase 2 field work	ARCADIS BBL	August 2007	October 2007 (approximate)	No	NA
Sample analysis, data processing and validation, and agency meetings	ARCADIS BBL	October 2007	March 2008 (approximate)	No	NA
Phase 3 field work	ARCADIS BBL	March 2008	April 2008 (approximate)	No	NA
Quarterly groundwater monitoring in the former Plainwell Impoundment monitoring wells	ARCADIS BBL	January 1, 2008	December 31, 2010	Yes	Two months after sampling event
SRI Report	ARCADIS BBL	NA	September 2011	Yes	NA
Area-Specific Alternatives Screening Technical Memorandum	ARCADIS BBL	NA	NA	Yes	60 days after draft SRI Report
FS Report	ARCADIS BBL	NA	NA	Yes	90 days after Specific Alternatives Screening Technical Memorandum comments

Note:
[1]Or as otherwise established in the Master Schedule for the Site.

QAPP Worksheet #17 Sampling Design and Rationale

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

TCRA Investigation

To facilitate confirmation sampling, a rectangular grid will be established within the excavated removal area as specified in the TCRA Design Report. The grid is simply for the purpose of systematically providing approximately 20 possible sampling points per area, from which five will be randomly selected and sampled. The exact size and shape of the grid is much less important than its use to verify that composite samples are representative of post-excavation PCB concentrations within the removal area. If the target area is smaller or irregular in shape, the overall number, shape, or size of individual grids may be modified, which can be done without diminishing the integrity, purpose, or results of the sampling approach. Five nodes from each grid will then be randomly selected for composite sampling (a random number generator/table or other means of selecting unbiased nodes will be used for choosing which nodes are sampled within each removal area sampling grid). The random pattern may be modified in the field (in consultation with oversight personnel) if excessive bias is present upstream, downstream, or laterally in the grid (i.e., sampling locations would ideally include nodes near/at the shoreline, near/at the bank and nodes in between).

The grid is preferable because it can be adapted to fit varying shoreline conditions. In addition, selecting five of the nodes provides that the composite sample is representative of the entire grid area.

Turbidity monitoring will be conducted upstream and downstream of the work area as specified in the TCRA Design Report. These locations were selected because, while the removal action will disturb the river in the immediate vicinity of the work, it is necessary to know what sorts of impacts (if any) this work is having on the rest of the river.

Former Plainwell Impoundment TCRA Sampling

PCBs in the sediments, river bank soils and floodplain soils will be sampled. The goal is to remove soils to a PCB concentration of 5 mg/kg or less, per composite sample. Extensive soil and sediment sampling has been conducted throughout the river. These results were used to determine the removal areas and serve as the background samples. The number of composite samples collected will vary based on shoreline size and shape. These samples will be collected at the conclusion of removal activities and should not be affected by seasonal considerations.

Turbidity will be monitoring hourly so that any disturbances to the river can be detected in real time and work can be altered as necessary.

QAPP Worksheet #17 Sampling Design and Rationale

SRI/FS Investigation for the Morrow Dam to Plainwell Area
The majority of the data needs from Morrow Dam to the Plainwell Dam and in the reach of Portage Creek included in the Work Plan are primarily related to characterizing the nature and extent of PCBs found within these areas, with additional data needed for a screening-level assessment of risks related to hazardous substances other than PCBs. These data needs will be fulfilled by collecting sediment, riverbank and floodplain soil, surface-water data and assessing the effects of the former Plainwell Impoundment Removal Action as described in the SRI/FS Work Plan for the Morrow Dam to Plainwell Area. The sediment data will be collected to further characterize the study area, update trend analyses and facilitate an assessment of risk for PCBs and potentially other constituents. The data collected will be of comparable quality to support statistical comparisons of PCB concentrations by various evaluations (i.e., through time and among reaches). Additionally, this sediment data will be collected to support future development, if needed, of both quantitative or qualitative sediment and constituent transport model. Riverbank and floodplain soil data will be collected to characterize the area historically inundated by the Plainwell No. 2 Dam and along the banks of the river and mill race from this dam to the area of the former Plainwell Inc. Mill. Surface-water data will be collected to evaluate effects of the removal action in the former Plainwell Impoundment on downstream transport of PCBs and solids remaining in the channel after implementation. Assessment of the effects of the former Plainwell Impoundment Removal Action will involve determining the rate of erosion of the mid-channel sediment prism that will be left behind (measured by bathymetry data collection), as well as determining the impact of construction activities on downstream solids and PCB transport (surface-water measurements). Additionally, PCB exposure concentrations in the Otsego City Impoundment will be quantified by monitoring yearling smallmouth bass tissue PCB concentrations in the Otsego City Impoundment after completing the removal action. A network of groundwater monitoring wells will be emplaced upon completion of the removal action as specified in the TCRA Design Report. This network was designed to include areas where groundwater discharges into the river and where high PCB concentrations were historically found. Groundwater sampling will be conducted quarterly for 2 years from 15 wells upon completion of all removal activities. Each sample will be analyzed for PCBs, TSS, TOC, TDS, chloride, sulfate, alkalinity, sodium, potassium, manganese and calcium. These parameters were selected because they represent the constituents historically found at the site. Concentrations will be compared to the MDEQ Residential Generic Cleanup Criteria and Screening Levels as described in Table 1 of MDEQ Remediation and Redevelopment Division (RRD) Operational Memorandum No. 1. The sampling design and rationale is described in the SRI/FS Work Plan as negotiated with the USEPA and other shareholders.

QAPP Worksheet #18-1 Sampling Locations and Methods/SOP Requirements (TCRA)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
TCRA Assessment Activities							
Resuspension Monitoring and Control Plan	Water	2 water samples weekly during construction (est. 16-wk period)	Total PCBs	Low	Approx 32 + 16 field duplicates	F-1	One grab water sample each will be collected mid-depth, mid-channel upstream (200 ft) and downstream (300 ft) of the work area
	Suspended solids in water column	Monitoring during construction hours[4]	Turbidity	Normal	Instantaneous readings conducted at 1-hour intervals at 3 locations		Readings taken at mid-depth, mid-channel upstream and two downstream locations
Dewatering Treatment System Discharges	Influent water	Weekly sampling at 5 outfalls during construction (est. 16-week period)	Total PCBs by USEPA Method 608	Low	Approx 90 + 16 field duplicates	F-1	One grab influent water sample will be collected at Outfalls 001, 002, 003, 004 and 005
	Intermediate-stage water	Weekly sampling at 5 outfalls during construction (est. 16-week period)	Total PCBs by USEPA Method 608	Low	Approx 90 + 16 field duplicates		One grab intermediate-stage water sample will be collected at Outfalls 001, 002, 003, 004 and 005
	Discharge water	Weekly sampling at 5 outfalls during construction (est. 16-week period)	Total PCBs by USEPA Method 608	Low	Approx 90 + 16 field duplicates	F-1	One grab discharge water sample will be collected at Outfalls 001, 002, 003, 004 and 005
		Weekly sampling at 5 outfalls during construction (est. 16-week period)	TSS by USEPA Method 160.2	Low	Approx 90 + 16 field duplicates		
		Monthly sampling at 5 outfalls during construction (est. 4-month period)	Total phosphorous as P by USEPA Method 365.3	Low	Approx 20 + 4 field duplicates		

QAPP Worksheet #18-1 Sampling Locations and Methods/SOP Requirements (TCRA)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
Post-Construction Confirmation Monitoring Within Removal Area — Near-Shore Area	Sediment	5 samples from each grid (3 to 6 inches below surface) for screening analysis	Total PCBs by screening method	High	5 minimum per grid + 1 field duplicate, but more if PCBs are detected by screening method	F-4 and F-6	Within each near-shore grid, 20 sample locations will be identified and 5 locations will be randomly selected for sampling; an additional 5 locations will be sampled if additional excavation is necessary
		1 composite sample from the 5 randomly selected samples	Total PCBs	Low	1 + 1 field duplicate, but more if PCB concentration is greater than 1 mg/kg	F-4	1 composite sample from the 5 randomly selected samples that pass screening criteria (<5 mg/kg PCB)
Post-Construction Confirmation Monitoring Within Removal Area — Mid-Channel Area	Sediment	5 samples from each of 3 mid-channel areas (3 to 6 inches below surface or deeper) for screening analysis	Total PCBs by screening method	High	15 minimum + 1 field duplicate, but more if PCBs are detected by screening method	F-4 and F-6	Within each mid-channel area, 20 sample locations will be identified and 5 locations will be randomly selected for sampling; an additional 5 locations will be sampled if additional excavation is necessary
		1 composite sample from the 5 randomly selected samples	Total PCBs	Low	3 + 3 field duplicate, but more if PCB concentration is greater than 1 mg/kg	F-5	1 composite sample from the 5 randomly selected samples that pass screening criteria (<5 mg/kg PCB) in each mid-channel area

QAPP Worksheet #18-1 Sampling Locations and Methods/SOP Requirements (TCRA)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
Post-Construction Confirmation Monitoring Within Removal Area — Bank Areas	Soil	5 samples from each grid (3 to 6 inches below surface) for screening analysis	Total PCBs by screening method	High	5 minimum per grid + 1 field duplicate, but more if PCBs are detected	F-4 and F-6	Within each bank grid, 20 sample locations will be identified and 5 locations will be randomly selected for sampling; an additional 5 locations will be sampled if additional excavation is necessary
		1 composite sample from the 5 randomly selected samples	Total PCBs	Low	1 + 1 field duplicate, but more if PCB concentration is greater than 5 mg/kg	F-5	1 composite sample from the 5 randomly selected samples that pass screening criteria (<5 mg/kg PCB)
Post-Construction Confirmation Monitoring Within Removal Area — Floodplain Area	Soil	5 samples from each grid (3 to 6 inches below surface) for screening analysis	Total PCBs by screening method	High	5 minimum per grid + field duplicate, but more if PCBs are detected	F-5 and F-6	Within each floodplain grid, 20 sample locations will be identified and 5 locations will be randomly selected for sampling; an additional 5 locations will be sampled if additional excavation is necessary
		1 composite sample from the 5 randomly selected samples	Total PCBs	Low	1 + 1 field duplicate, but more if PCB concentration is greater than 5 mg/kg	F-5	1 composite sample from the 5 randomly selected samples that pass screening criteria (<5 mg/kg PCB)

QAPP Worksheet #18-1 Sampling Locations and Methods/SOP Requirements (TCRA)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
Backfill Soil Taken From Former Plainwell Impoundment Area	Soil	1 composite sample for each 10,000 cubic yards	TCL — VOCs	Low	1 + 1 field duplicate for each 10,000 cubic yards	F-5	Composite sample consists of 9 subsamples from various locations of the backfill volume
			TCL — SVOCs	Low	1 + 1 field duplicate		
			TCL — Pesticides/PCBs	Low	1 + 1 field duplicate		
			RCRA metals	Low	1 + 1 field duplicate		
			TPH-GRO	Low	1 + 1 field duplicate		
			TPH-DRO	Low	1 + 1 field duplicate		
Post-Removal Monitoring Within Removal Area	Sediment	39 surface sediment samples (0-2 inch)	Total PCBs	Low	39 + 2 field duplicates	F-4	At each of the 13 removal areas (Removal Area 1 through 13), surficial sediment samples will be collected at 3 locations, equally spaced in the area
			TOC	Normal	39 + 2 field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	39 + 2 field duplicates		
	Groundwater	15 monitoring well water samples per quarter for 2 years	Total PCBs	Low	15 + 1 field duplicate per quarter — 128 total	F-10	At each well (monitoring wells 1 through 15), one water sample will be collected each quarter for 2 years
			TOC	Low	15 + 1 field duplicate per quarter — 128 total		
			TDS	Low	15 + 1 field duplicate per quarter — 128 total		
			TSS	Low	15 + 1 field duplicate per quarter — 128 total		
			Chloride	Low	15 + 2 field duplicates per quarter — 128 total		

QAPP Worksheet #18-1 Sampling Locations and Methods/SOP Requirements (TCRA)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
Post-Removal Monitoring Within Removal Area	Groundwater	15 monitoring well water samples per quarter for 2 years	Sulfate	Low	15 + 1 field duplicate per quarter — 128 total		
			Alkalinity	Low	15 + 1 field duplicate per quarter — 128 total		
			Sodium	Low	15 + 1 field duplicate per quarter — 128 total		
			Potassium	Low	15 + 1 field duplicate per quarter — 128 total		
			Magnesium	Low	15 + 1 field duplicate per quarter — 128 total		
			Calcium	Low	15 + 1 field duplicate per quarter — 128 total		

Notes:
1. One blind duplicate sample will be collected for every 20 samples.
2. Sampling SOP reference number from QAPP Worksheet #21.
3. Supplemental turbidity monitoring may be necessary to identify potential problems with the resuspension control system.

All PCBs are analyzed by Method 8082 except where noted.

QAPP Worksheet #18-2 Sampling Locations and Methods/SOP Requirements (SRI/FS)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
SRI/FS Activities							
Reach #1: Georgia-Pacific Mill to Crown Vantage — Phase 1	Sediment/Soil	80 cores	NA	NA	NA	F-4/F-5	10 transects, 8 locations/transect —including top-of-bank locations for sediment and soil characterization
Reach #1: Georgia-Pacific Mill to Crown Vantage — Phase 2	Sediment	30 cores[3]: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to bottom	PCBs	Low	120 + 6 field duplicates	F-4	3 cores per transect
			TOC	Normal	120 + 6 field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	120 + 6 field duplicates		
		2 cores: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to bottom	PCBs	Low	8 + 1 field duplicate	F-4	2 cores randomly selected from the 30 cores (above)
			VOCs	Low	8 + 1 field duplicate		
			SVOCS	Low	8 + 1 field duplicate		
			Chlorinated pesticides	Low	8 + 1 field duplicate		
			TAL metals	Low	8 + 1 field duplicate		
			TOC	Normal	8 + 1 field duplicate		
			Particle size	Cobble, sand, silt, clay fractions	8 + 1 field duplicate		
		2 cores: surficial interval only	AVS/SEM	Low	2 + 1 field duplicate		

QAPP Worksheet #18-2 Sampling Locations and Methods/SOP Requirements (SRI/FS)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
Reach #1: Georgia-Pacific Mill to Crown Vantage — Phase 2 Agency-Directed Core Collection	Sediment	20 cores: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to core bottom	Total PCBs	Low	80 + 4 field duplicates	F-4	Cores collected at 20 agency-directed locations in this reach
			TOC	Normal	80 + 4 field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	80 + 4 field duplicates		
Reach #1: Georgia-Pacific Mill to Crown Vantage — Phase 2 Resample of 2000 Locations	Sediment	26 cores: surficial sample (0 to 2 inch) only	Total PCBs	Low	67 + 3 field duplicates	F-4	See Table 3-4 of Work Plan for specific sample locations
			TOC	Normal	67 + 3 field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	67 + 3 field duplicates		
Reach #1: Georgia-Pacific Mill to Crown Vantage — Phase 3 'Hot Spot' Assessment	Sediment	42 cores: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to core bottom	Total PCBs	Low	168 + 9 field duplicates	F-4	Based on Phase 2, six to eight locations will be selected and approved by USEPA and 4 to 8 cores analyzed around each location
			TOC	Normal	168 + 9 field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	168 + 9 field duplicates		
Reach #2: Crown Vantage to Plainwell No. 2 Dam — Phase 1 Focused Sampling	Sediment/Soil	25 cores: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to core bottom	Total PCBs	Low	100 + 5 field duplicates	F-4/F-5	Return to sample locations FF-32, FF-35, FF-37, FF-40, FF-42; collect 4 to 8 cores at each location
			TOC	Normal	100 + 5 field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	100 + 5 field duplicates		
Reach #3: Plainwell No. 2 Dam to Mill Race Confluence — Phase 1	Sediment/Soil	60 cores	NA	NA	NA	F-4/F-5	6 transects, 10 locations/transect — including top-of-bank locations for sediment and soil characterization.

QAPP Worksheet #18-2 Sampling Locations and Methods/SOP Requirements (SRI/FS)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
Reach #3: Plainwell No. 2 Dam to Mill Race Confluence — Phase 2 Transect Sediment/Soil Analysis	Sediment/Soil	30 cores: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to core bottom	Total PCBs	Low	120 + 6 field duplicates	F-4/F-5	Select 5 cores per transect (6 transects)
			TOC	Normal	120 + 6 field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	120 + 6 field duplicates		
		2 cores: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to bottom	PCBs	Low	8 + 1 field duplicate	F-4/F-5	2 sediment cores randomly selected from the 30 cores (above)
			VOCs	Low	8 + 1 field duplicate		
			SVOCs	Low	8 + 1 field duplicate		
			Chlorinated pesticides	Low	8 + 1 field duplicate		
			TAL metals	Low	8 + 1 field duplicate		
			TOC	Normal	8 + 1 field duplicate		
			Particle size	Cobble, sand, silt, clay fractions	8 + 1 field duplicate		
		2 cores: surficial interval only	AVS/SEM	Low	2 + 1 field duplicate		
Reach #3: Plainwell No. 2 Dam to Mill Race Confluence — Phase 2 Resample of 2000 Locations	Sediment	18 cores: surficial sample (0 to 2 inch) only	Total PCBs	Low	18 + 1 field duplicates	F-4	See Table 3-4 of Work Plan for specific sample locations
			TOC	Normal	18 + 1 field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	18 + 1 field duplicates		

QAPP Worksheet #18-2 Sampling Locations and Methods/SOP Requirements (SRI/FS)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
Reach #3: Plainwell No. 2 Dam to Mill Race Confluence — Phase 3 'Hot Spot' Assessment	Sediment	12 cores: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to core bottom	Total PCBs	Low	48 + 3 field duplicates	F-4	Based on Phase 2, one to three locations will be selected and approved by USEPA and 4 to 8 cores analyzed around each location
			TOC	Normal	48 + 3 field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	48 + 3 field duplicates		
Reach #4: Portage Creek — Phase 1 Mapping	Sediment/Soil	NA	NA	NA	NA	F-4	Mapping of sediment deposits from Alcott Street to Kalamazoo River, including floodplain areas.
Reach #4: Portage Creek — Phase 2 Core Collection	Sediment	40 cores: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to core bottom	Total PCBs	Low	160 + 8 field duplicates	F-4	40 cores will be collected, and approved by USEPA
			TOC	Normal	160 + 8 field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	160 + 8 field duplicates		
		2 cores: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to bottom	PCBs	Low	8 + 1 field duplicate	F-4	2 sediment cores randomly selected from the 40 cores (above)
			VOCs	Low	8 + 1 field duplicate		
			SVOCS	Low	8 + 1 field duplicate		
			Chlorinated pesticides	Low	8 + 1 field duplicate		
			TAL metals	Low	8 + 1 field duplicate		
			TOC	Normal	8 + 1 field duplicate		
			Particle Size	Cobble, sand, silt, clay fractions	8 + 1 field duplicate		

QAPP Worksheet #18-2 Sampling Locations and Methods/SOP Requirements (SRI/FS)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
Reach #4: Portage Creek — Phase 2 Core Collection		2 cores: surficial interval only	AVS/SEM	Low	2 + 1 field duplicate		
Reach #4: Portage Creek — Phase 3 'Hot Spot' Assessment, if needed	Sediment	TBD cores: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to core bottom	Total PCBs	Low	TBD + TBD field duplicates	F-4	Based on Phase 2 and determined by USEPA, locations are selected and 4 to 8 cores analyzed around each location
			TOC	Normal	TBD + TBD field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	TBD + TBD field duplicates		
Reach #5: Crown Vantage Area — Phase 1 Field Reconnaissance	Sediment	NA	NA	NA	NA	F-4	Delineate waste in side channel of Crown Vantage Site
Reach #5: Crown Vantage Area — Phase 2 Characterization, if needed	Sediment	TBD cores: 0 to 2 inch, 2 to 6 inch, 6 to 12, 1 foot to core bottom	Total PCBs	Low	TBD + TBD field duplicates	F-4	Based on Phase 1 results and determined by USEPA, TBD locations are selected for sampling
			TOC	Normal	TBD + TBD field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	TBD + TBD field duplicates		
Area of Historical Inundation near Plainwell No. 2 Dam —Phase 1 Floodplain Sampling	Soil	TBD cores: 0 to 6 inch, 6 to 12, 1 foot to core bottom	Total PCBs	Low	TBD + TBD field duplicates	F-5	Map extent of inundated area and characterize soil/vegetation; based on observations, select soil cores with approval from the USEPA
			TOC	Normal	TBD + TBD field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	TBD + TBD field duplicates		

QAPP Worksheet #18-2 Sampling Locations and Methods/SOP Requirements (SRI/FS)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
Area of Historical Inundation near Plainwell No. 2 Dam —Phase 2 Radial, Grid-like Sampling, if necessary	Soil	TBD cores: 0 to 6 inch, 6 to 12, 1 foot to core bottom	Total PCBs	Low	TBD + TBD field duplicates	F-5	Based on Phase 1, if necessary, cores will be collected in grid-like pattern with approval from the USEPA
			TOC	Normal	TBD + TBD field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	TBD + TBD field duplicates		
Area Along Banks Between Plainwell No. 2 Dam and Mill Race Confluence	Soil	12 cores: 0 to 6 inch, 6 to 12, 1 foot to core bottom	Total PCBs	Low	36 + 2 field duplicates	F-5	Bank soils collected at each end of 6 sediment transects established in Reach #3 above
			TOC	Normal	36 + 2 field duplicates		
			Particle size	Cobble, sand, silt, clay fractions	36 + 2 field duplicates		
Assessment of Effects from the former Plainwell Impoundment Area	Bathymetry	NA	NA	NA	NA	F-4	10 transects will be survey before, during, and for 2 years after (2 per year) the TCRA
	Water	2 sets of samples every other day during construction	PCB Aroclors	Low	TBD + TBD field duplicates	F-1	1 set of depth-integrated samples will be collected upstream at Main Street Bridge in Plainwell, and 1 set of time-integrated samples (24 hourly samples taken with ISCO sampler) then composited, collected downstream at the Farmer Street Bridge in Otsego
			TSS	Normal	TBD + TBD field duplicates		

QAPP Worksheet #18-2 Sampling Locations and Methods/SOP Requirements (SRI/FS)

Sampling Location/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
Groundwater Monitoring	Water	Coordinate monitoring (2 years) quarterly for 15 wells (8 X 15)	Total PCBs (Aroclor), Total Organic Carbon, Total Dissolved Solids, Total Suspended Solids, Chloride, Sulfate, Alkalinity, Sodium, Potassium, Magnesium, Calcium	Low	128	F-9/F-10	Coordinate samples will be used to evaluate the presence of PCB in the post-removal impoundment.
Otsego City Impoundment Area (Aquatic Biota Sampling Area 6)	Whole fish	7 composite samples of 5 yearling smallmouth bass (whole-body) each will be collected in fall 2006, 2007, and 2008, or until construction is complete.	Total PCBs	Low	21	F-7	During and after construction in 2007 and 2008, yearling smallmouth bass will be collected for analysis
			Lipids	Low	21		
			Weight and length of each fish	Normal	105		

Notes:
1. One blind duplicate sample will be collected for every 20 samples.
2. Reference number from QAPP Worksheet #21.
3. Based on PCB results of these 30 cores, up to 20 cores (2 more cores per transect on average) will be selected for similar sectioning and analyses.

All PCBs are analyzed by Method 8082 except where noted.

QAPP Worksheet #19 Analytical SOP Requirements (Sample Containers, Preservation and Holding Times)

Parameter	Analytical and Preparation Method/SOP Reference	Method	Bottle Type	Preservation	Holding Time
Water (Discharge Monitoring)					
PCB	SOP KO8082.09. Also, SOP KO3510, SOP KO3500B and SOP KO3665A.	608 [6]	Two 1-L amber glass bottle with Teflon®-lined lid	Cool to 4°C±2°C	7 days to extraction 40 days to analysis
TSS	SOP KQ468.04	160.2 [1]	One 1-L plastic bottle	Cool to 4°C±2°C	7 days to analysis
Total Phosphorus as P	SOP KQ450.05	365.2 [1]	500ml plastic bottle	H_2SO_4 to pH<2, Cool to 4°C	28 days to analysis
Water (Groundwater-Surface)					
PCB	SOP LM-GC-8082. Also, SOPs LM-OP-3510 and LM-OP-Cleanup	8082 [1]	Two 1-L amber glass bottle with Teflon®-lined lid	Cool to 4°C±2°C	7 days to extraction 40 days to analysis
Metals (Na, K, Mg, Ca)	SOP LM-MI-6010B	6010 [1]	One 500ml plastic bottle	HNO_3 to pH<2, Cool to 4°C±2°C	180 days to analysis
TOC	SOP LM-WC-415.1/9060	9060 [1]	Two 40-ml glass vial	H_2SO_4 to pH<2, Cool to 4°C	28 days to analysis
TDS	SOP LM-WC-160.1	160.1 [1]	One 1-L plastic bottle	Cool to 4°C±2°C	7 days to analysis
TSS	SOP LM-WC-160.2	160.2 [1]			7 days to analysis
Chloride	SOP KQ600E.04	300 [1]	One 1-L plastic bottle		28 days to analysis
Sulfate	SOP KQ600E.04	300 [1]			28 days to analysis
Alkalinity	SOP KQ120.04	310.2 [1]			14 days to analysis
Soil (TCRA Backfill)					
VOCs	SOP LM-MV-8260B	8260 [2]	3-EnCore™ samplers One 40-ml glass vial	Cool to 4°C±2°C	48 hours to preservation 14 days to analysis
SVOCs	SOP LM-MS-8270C. Also, SOP LP-OP-3541	8270 [2]	One 8-oz glass jar with Teflon®-lined lid	Cool to 4°C±2°C	14 days to extraction 40 days to analysis
PCB	SOP LM-GC-8082. Also, SOPs LM-OP-3541, LM-OP-3660B, and LM-OP-Cleanup	8082 [2]			14 days to extraction 40 days to analysis
Pesticides	SOP LM-GC-8081. Also, SOP LP-OP 3541	8081 [2]	One4-oz glass jar with Teflon®-lined lid	Cool to 4°C±2°C	14 days to extraction 40 days to analysis
TPH (DRO)	SOP KO3545	8270 [2]	One4-oz glass jar with Teflon®-lined lid		14 days to extraction 40 days to analysis
TPH (DRO and GRO)	SOP KO3545	8260 [2]	3-EnCore™ samplers One 40-ml glass vial	Cool to 4°C±2°C	14 days to extraction 40 days to analysis
Metals (except mercury)	SOP LM-MI-6010B. Also, SOP LM-MP-3050	6010 [2]	One 4-oz wide-mouth glass jar	Cool to 4°C±2°C	180 days to analysis
Mercury	SOP LM-HG-7471A	7471 [2]			28 days to analysis
Soil/Sediments (SRI/FS)					
VOCs	SOP LM-MV-SOM01.1-VOA	CLP SOM01.1[3,7]	3-EnCore™ samplers One 40-ml glass vial	Cool to 4°C±2°C	48 hours to preservation 10 days to analysis
SVOCs	SOP LM-MS-SOM01.1-SVOA	CLP SOM01.1[3,7]	One 8-oz glass jar with Teflon®-lined lid	Cool to 4°C±2°C	5 days to extraction 40 days to analysis
PCB	SOP LM-GC-8082. Also, SOPs LM-OP-3541, LM-OP-3660B, and LM-OP-Cleanup	8082 [6]			14 days to extraction 40 days to analysis
Pesticides	SOP LM-GC-SOM01.1-PEST	CLP SOM01.1[3,7]	One 8-oz glass jar with Teflon®-lined lid	Cool to 4°C±2°C	5 days to extraction 40 days to analysis

QAPP Worksheet #19 Analytical SOP Requirements (Sample Containers, Preservation and Holding Times)

Parameter	Analytical and Preparation Method/SOP Reference	Method	Bottle Type	Preservation	Holding Time
Soil/Sediments (SRI/FS) (Cont'd.)					
Metals (except mercury)	SOP LM-MI-ILM05.3-ICP-AES	CLP ILM05.3[4,7]	One 4-oz wide-mouth glass jar	Cool to 4°C±2°C	180 days to analysis
Mercury					28 days to analysis
Acid Volatile Sulfides/Simultaneously Extracted Metals (AVS/SEM)	SOP LM-WC-AVS/SEMSOP LM-MI-6010B and LM-HG-7470	AVS/SEM	One 4-oz wide-mouth glass jar	Cool to 4°C±2°C	180 days to analysis
					28 days to analysis
Particle size	LM-SL-D422	ASTM D422-63	large Ziploc® bag	NS	NS
TOC	SOP LM-WC-TOC LK	Lloyd Kahn[5]	One 4-oz wide-mouth glass jar	Cool to 4°C±2°C	14 days to analysis
Fish (Otsego City Impoundment)					
PCB	SOP LM-GC-8082. Also, SOPs LM-Tissue, and LM-OP-Cleanup	8082 [6]	Wrap with aluminum foil	In the Field Cool to 4°C+2°C, Laboratory will Cool to -15°C+2°C upon receipt	7 days to extraction following removal from frozen storage
					40 days to analysis
Lipids	SOP LM-OP-Lipids	STL Lipids[8]			7 days to analysis following removal from frozen storage

Notes:

1. USEPA. *Methods for Chemical Analysis of Water and Wastes*. EPA/600/4-79/020. EMSL-Cincinnati. 1983.
2. USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846.* 3rd ed. Washington, DC. 1996.
3. USEPA. *Contract Laboratory Program Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration*, SOM01.1. May 2005.
4. USEPA. *Contract Laboratory Program Statement of Work For Inorganic Analysis, Multi-Media, Multi-Concentration*, ILM05.3. March 2004.
5. USEPA. *Determination of Total Organic Carbon in Sediment (Lloyd Kahn Method).* July 27, 1988.
6. USEPA. *Appendix A to Part 136 Methods For Organic Chemical Analysis of Municipal and Industrial Wastewater Method 608—Organocholrine Pesticides and PCBs.* July 1995.
7. All holding times are measured from date of collection with the exception of CLP methods which are based on Validated Time of Sample Receipt (VTSR).
8. STL Standard Operating Procedure for percent lipids determination.

°C = Degrees Celsius.
mL/L = Milliliters per liter.
PCB = Polychlorinated biphenyl.
NS = Not Specified.
TOC = Total organic carbon.
TDS = Total dissolved solid.
TSS = Total suspended solid.

QAPP Worksheet #20 Sample Quantities and Control Frequencies

	Laboratory	Analytical and Preparation SOP[1]	Estimated Environ. Sample Quality	Field QC Analyses						Laboratory QC Sample						Total
				Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Lab Duplicate		
				Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
TCRA																
Water (Resuspension Monitoring and Control Plan)																
PCB (USEPA 608)	KAR[5]	SOP KO8082.09. Also, SOP KO3510, SOP KO3500B and SOP KO3665A.	TBD	NA	--	1/day	--	1/20	TBD	1/20	--	1/20	--	NA	--	TBD
Water (Dewatering System Discharge Monitoring Plan)																
PCB (USEPA 608)	KAR[5]	SOP KO8082.09. Also, SOP KO3510, SOP KO3500B and SOP KO3665A.	TBD	NA	--	1/day	--	1/20	TBD	1/20	--	1/20	--	NA	--	TBD
TSS (USEPA 160.2)	KAR[5]	SOP KQ468.04	TBD	NA	--	1/day	--	1/20	TBD	NA	--	NA	--	1/20	--	TBD
Total Phosphorus as P (USEPA 365.2)	KAR[5]	SOP KQ450.05	TBD	NA	--	1/day	--	1/20	TBD	NA	--	NA	--	1/20	--	TBD
Near-Shore Sediment Post-Removal Confirmation Monitoring																
PCB (Immunoassay)	NA	Field Screening for PCBs Using Strategic Diagnostics, Inc. EnSYS Corp. PCB Soil and Wipe Test System	TBD	NA	--	NA	--	1/20	TBD	NA	--	NA	--	NA	--	TBD
PCB (SW-846 8082)	KAR[5]	SOP KO8082.09. Also, SOP KO3545, SOP KO3500B and SOP KO3665A	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
Mid-Channel Sediment Post-Removal Confirmation Monitoring																
PCB (Immunoassay)	NA	Field Screening for PCBs Using Strategic Diagnostics, Inc. EnSYS Corp. PCB Soil and Wipe Test System	TBD	NA	--	NA	--	1/20	TBD	NA	--	NA	--	NA	--	TBD
PCB (SW-846 8082)	KAR[5]	SOP KO8082.09. Also, SOP KO3545, SOP KO3500B and SOP KO3665A	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
Bank Soil PCB Concentration Confirmation Monitoring																
PCB (Immunoassay)	NA	Field Screening for PCBs Using Strategic Diagnostics, Inc. EnSYS Corp. PCB Soil and Wipe Test System	TBD	NA	--	NA	--	1/20	TBD	NA	--	NA	--	NA	--	TBD
PCB (SW-846 8082)	KAR[5]	SOP KO8082.09. Also, SOP KO3545, SOP KO3500B and SOP KO3665A	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD

QAPP Worksheet #20 Sample Quantities and Control Frequencies

	Laboratory	Analytical and Preparation SOP[1]	Estimated Environ. Sample Quality	Field QC Analyses						Laboratory QC Sample						Total
				Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Lab Duplicate		
				Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Floodplain Soil PCB Concentration Confirmation Monitoring																
PCB (Immunoassay)	NA	Field Screening for PCBs Using Strategic Diagnostics, Inc. EnSYS Corp. PCB Soil and Wipe Test System	TBD	NA	--	NA	--	1/20	TBD	NA	--	NA	--	NA	--	TBD
PCB (SW-846 8082)	KAR[5]	SOP KO8082.09. Also, SOP KO3545, SOP KO3500B and SOP KO3665A	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
Backfill Soil Sampling																
VOCs (SW-846 8260)	STL[6]	SOP LM-MV-8260B	TBD	1/cooler	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
SVOCs (SW-846 8270)	STL[6]	SOP LM-MS-8270C. Also, SOP LP-OP-3541	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
Chlorinated Pesticides (SW-846 8081)	STL[6]	SOP LM-GC-8081. Also, SOP LP-OP 3541	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
RCRA Metals	STL[6]	SOP LM-MI-6010B and SOP LM-HG-7471. Also, SOP LM-MP-3050	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	NA	--	1/20	TBD	TBD
PCB (SW-846 8082)	STL[6]	SOP LM-GC-8082. Also, SOPs LM-OP-3541, LM-OP-3660B, and LM-OP-Cleanup	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
TPH (DRO)	KAR[5]	SOP KO8270-DRO.05. Also, SOP KO3545	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
TPH (GRO)	KAR[5]	SOP KOTPH-GROHP.03	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
Post-Construction Sediment Sampling																
PCB (SW-846 8082)	STL[6]	SOP LM-GC-8082. Also, SOPs LM-OP-3541, LM-OP-3660B, and LM-OP-Cleanup	39	NA	--	1/day	2	1/20	2	1/20	2	1/20	2	NA	--	47
TOC (Lloyd Kahn)	STL[6]	SOP LM-WC-TOC LK	39	NA	--	1/day	2	1/20	2	1/20	2	NA	--	1/20	2	47
Particle Size	STL[6]	SOP LM-SL-D422	39	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	39
Water (Groundwater/Surface-Water Interface Monitoring Program)																
PCB (SW-846 8082)	STL[6]	SOP LM-GC-8082. Also, SOPs LM-OP-3510 and LM-OP-Cleanup	15	NA	--	1/day	1	1/20	1	1/20	1	1/20	1	NA	--	19
Total Organic Carbon (9060)	STL[6]	SOP LM-WC-415.1/9060	15	NA	--	1/day	1	1/20	1	1/20	1	NA	--	1/20	1	19
TDS (USEPA 160.1)	STL[6]	SOP LM-WC-160.1	15	NA	--	1/day	1	1/20	1	NA	--	NA	--	1/20	1	18
TSS (USEPA 160.2)	STL[6]	SOP LM-WC-160.2	15	NA	--	1/day	1	1/20	1	NA	--	NA	--	1/20	1	18
Chloride (USEPA 300)	KAR[5]	SOP KQ600E.04	15	NA	--	1/day	1	1/20	1	1/20	1	NA	--	1/20	1	19
Sulfate (USEPA 300)	KAR[5]	SOP KQ600E.04	15	NA	--	1/day	1	1/20	1	1/20	1	NA	--	1/20	1	19
Alkalinity (USEPA 310.1)	KAR[5]	SOP KQ120.04	15	NA	--	1/day	1	1/20	1	1/20	1	NA	--	1/20	1	19
Metals (Na, K, Mg, Ca) (SW-846 6010)	STL[6]	SOP LM-MI-6010B	15	NA	--	1/20	1	1/20	1	1/20	1	NA	--	1/20	1	19

QAPP Worksheet #20 Sample Quantities and Control Frequencies

	Laboratory	Analytical and Preparation SOP[1]	Estimated Environ. Sample Quality	Field QC Analyses						Laboratory QC Sample						Total
				Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Lab Duplicate		
				Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
SRI/FS																
Sediment																
PCB (SW-846 8082)	STL[6]	SOP LM-GC-8082. Also, SOPs LM-OP-3541, LM-OP-3660B, and LM-OP-Cleanup	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	--	1/20	--	NA	--	TBD
VOCs (CLP SOM01.1)	STL[6]	SOP LM-MV-SOM01-VOA	TBD	1/cooler	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
SVOCs (CLP SOM01.1)	STL[6]	SOP LM-MS-SOM01.1-SVOA	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
Chlorinated Pesticides (CLP SOM01.1)	STL[6]	SOP LM-GC-SOM01.1-PEST	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--	TBD
TAL Metals (CLP ILM05.3)	STL[6]	SOP LM-MI-ILM05.3-ICP-AES	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	NA	--	1/20	TBD	
AVS/SEM Metals (SW-846 6010)	STL[6]	SOP LM-WC-AVS/SEM. Also, SOPs LM-MI-6010B and LM-HG-7470	TBD	NA	--	1/day	TBD	1/20	TBD	1/20	TBD	NA	--	1/20	TBD	TBD
TOC (Lloyd Kahn)	STL[6]	SOP LM-WC-TOC LK	TBD	NA	--	1/day	--	1/20	TBD	1/20	TBD	NA	--	1/20	TBD	TBD
Particle Size	STL[6]	SOP LM-SL-D422	TBD	--	--	--	--	--	--	--	--	--	--	--	--	TBD
River Bank and Floodplain Soil Data																
PCB (SW-846 8082)	STL[6]	SOP LM-GC-8082. Also, SOPs LM-OP-3541, LM-OP-3660B, and LM-OP-Cleanup	TBD	NA	--	1/day	--	1/20	TBD	1/20	--	1/20	--	NA	--	TBD
TOC	STL[6]	SOP LM-WC-TOC LK	TBD	NA	--	1/day	--	1/20	TBD	1/20	TBD	NA	--	1/20	TBD	TBD
Particle Size	STL[6]	SOP LM-SL-D422	TBD	--	--	--	--	--	--	--	--	--	--	--	--	TBD
Surface-Water Data (Plainwell Impoundment)																
PCB (SW-846 8082)	STL[6]	SOP LM-GC-8082. Also, SOPs LM-OP-3510 and LM-OP-Cleanup	TBD	NA	--	1/day	--	1/20	TBD	1/20	--	1/20	--	NA	--	TBD
TSS (USEPA 160.2)	STL[6]	SOP LM-WC-160.2	TBD	NA	--	1/day	--	1/20	TBD	NA	--	NA	--	1/20	--	TBD
Fish (Otsego City Impoundment)																
PCB (SW-846 8082)	STL[6]	SOP LM-GC-8082. Also, SOPs LM-OP-3540, LM-OP-Cleanup, and LM-OP-GPC	TBD	NA	--	1/day	--	1/20	TBD	1/20	--	1/20	--	NA	--	TBD
Lipids	STL[6]	SOP LM-OP-Lipids	TBD	NA	--	--	--	--	--	--	--	--	--	1/20	TBD	TBD

Notes:
1. See WS#23 for SOP title, revision number, date details.
2. Sample counts are an approximation.
3. Field duplicates will consist of co-located samples for air analysis.
4. See Worksheet #30 for contact information.
5. Analysis to be performed by KAR.
6. Analysis to be performed by STL.

1/day = One rinse blank per day or one per 20 samples, whichever is more frequent. Rinse blanks not required when dedicated sampling equipment is used.
Freq = Frequency.
No. = Number.

QAPP Worksheet #21 Field Sampling SOP References

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
F-1	*FSP — Water Column Sampling Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes water column sampling procedures and the required equipment
F-2	*FSP — River Flow Measurement Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes the procedure for river flow measurement and the required equipment
F-3	*FSP— Water Quality Measurement Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes water quality measurement procedures and the required equipment
F-4	*FSP — Sediment Sampling Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes the procedures for sediment sampling and the required equipment
F-5	*FSP — Soil Sampling Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes the procedures for soil sampling and the required equipment
F-6	*FSP — Field Screening Using EnSys Corp. PCB Soil and Wipe Test System*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes use of the EnSys™ PCB Soil and Wipe Test System (by Strategic Diagnostics, Inc.) immunoassay field kit
F-7	*FSP — Fish Sampling Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes the procedures for fish sampling and the required equipment
F-8	*FSP— Fish Sample Processing and Handling Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes the procedure for fish sample processing and handling

QAPP Worksheet #21 Field Sampling SOP References

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
F-9	*FSP— Monitoring Well Installation and Development Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes the procedure for monitoring well installation and development; also describes the equipment associated with the procedure
F-10	*FSP— Groundwater Sampling Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes the procedure for groundwater sampling and the required equipment
F-11	*FSP — Field Equipment Cleaning and Decontamination Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes the procedure for field equipment cleaning and decontamination
F-12	*FSP — Field Samples Handling, Packing, and Shipping Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes field sample handling, packaging and shipping procedures
F-13	*FSP — Investigation Derived Waste Sampling Procedures*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes the procedure for Derived Waste sampling and the required equipment
F-14	*FSP — Freezing Procedures for Preserving PCBs in Sediments and Biota Samples*	ARCADIS BBL	See SOP for specific equipment needs	N	Describes the procedure for Preserving PCBs in Sediments and Biota and the required equipment

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

Field Equipment	Calibration Activity/ Frequency	Maintenance Activity	Testing Activity	Inspection Activity	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Combination Water Quality Meter	F-1	As required by manufacturer specifications	Temperature, conductivity, pH, turbidity, DO	Check all membranes and sensors, cable and check battery charge	F-1	F-1	F-1	F-1
Digital Camera	NA	NA	NA	Batteries charge, acceptable memory and general camera function	NA	NA	NA	F-4 and F-5
GPS	NA	As required by manufacturer specification	Coordinates	Batteries charge, acceptable memory	NA	NA	NA	F-4 and F-5
pH Meter	pH meter calibration will typically include two pH buffers bracketing expected pH range of samples to be measured (i.e. 7.00 and 4.00), with a verification of the slope using a third buffer (4.00 or 10.00)	As required by manufacturer specification	pH	Check membranes, battery charge	pH +/- 0.01 S.U.	Recalibrate pH meter	F-4	F-4

Note:
[1]SOPs are found in the Field Sampling Plan (FSP).

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
TCRA and SRI/FS						
F-1	FSP– Water Column Sampling Procedures	Definitive	Temperature, pH, turbidity, conductivity, DO	Combination water quality meter	ARCADIS BBL Field Team	N
F-4	FSP –Sediment Sampling Procedures	Screening	Water surface elevation	Staff gauges	ARCADIS BBL Field Team	N
F-4	FSP–Sediment Sampling Procedures	Screening	Core characterization	USGS method	ARCADIS BBL Field Team	N
F-6	FSP – Field Screening Using EnSys Corp. PCB Soil and Wipe Test System	Screening	PCB concentration	EnSYS Corp. PCB risk immunoassay, spectrophoto-meter	ARCADIS BBL Field Team	N
F-10	FSP– Groundwater Sampling Procedures	Definitive	Turbidity, temperature, specific conductance, pH	YSI 6-Series multiparameter instrument	ARCADIS BBL Field Team	N
F-10	FSP – Groundwater Sampling Procedures	Screening	Water surface elevation	Staff gauge	ARCADIS BBL Field Team	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
TCRA						
K-1	This SOP contains Method 608 methodology--'Analysis of PCB by EPA Method 8082', SOP KO8082.09, Dec. 19, 2006; also, SOP KO3510, SOP KO3500B and SOP KO3665A (sulfuric acid cleanup)	Definitive	Total PCB in water	Gas chromatography/ electron capture detectors (GC/ECD)	KAR	N
K-2	'Analysis of PCB by EPA Method 8082', SOP KO8082.09, Dec 19, 2006; also, SOP KO3545, SOP KO3500B and SOP KO3665A (sulfuric acid cleanup)	Definitive	Total PCB in sediment	GC/ECD	KAR	N
K-3	'Gravimetric Determination of Total, Fixed and Volatile Suspended Solids', SOP KQ468.04, May 30, 2006	Definitive	TSS	Scale	KAR	N
K-4	'Colorimetric Determination of Various Forms of Phosphorus', SOP KQ450.05, June 28,2006	Definitive	Total phosphorus as P in water	Spectrophotometer	KAR	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
K-5	'Diesel Range Organics - Total Petroleum Hydrocarbons by GC/MS (Method 8270)', SOP KO8270.05, December 19, 2005; also, SOP KO3545 (extraction)	Definitive	TPH diesel-range organics	Gas Chromatogram/ Mass Spectrometry (GC/MS)	KAR	Y
K-6	'Determination of Gasoline Range Organics by GC/MS by 8260B using HP GC-MS', SOP KOTPH-GROHP.04.' March 27, 2007	Definitive	TPH gasoline-range organics	GC/MS	KAR	Y
K-7	'Determination of Fluoride, Chloride, Nitrite, Nitrate, Bromide, Phosphate and/or Sulfate in Water by Ion Chromatography', SOP No.: KQ600E.04, October 10, 2006	Definitive	Chloride, sulfate in water	*Lachat QuikChem 8000 Ion Chromatograph*, Zellweger/Lachat	KAR	N
K-8	'Quantitation of Alkalinity in Water by Titration', SOP No.: KQ120.04, December 15, 2005	Definitive	Alkalinity in water	pH meter	KAR	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
S-1	'Volatile Organic Compounds by GC/MS SW-846 8260B', SOP LM-MV-8260B, Rev 5, December 12, 2005	Definitive	VOCs in soil	GC/MS	STL	N
S-2	'Semivolatile Organic Compounds by GC/MS SW-846 8270C', SOP LM-MS-8270C, Rev 5, December 12, 2005; also, SOP LP-OP-3541 (extraction)	Definitive	SVOCs in soil	GC/MS	STL	N
S-3	'Organochlorine Pesticides by Gas Chromatography SW-846 Method 8081A & EPA TO-4A; SOP LM-GC-8081' Rev 7, July 27, 2007; also, SOP LP-OP 3541 (extraction)	Definitive	Chlorinated pesticides in soil	GC/ECD	STL	N
S-4	'Inductively Coupled Plasma-Atomic Emission Spectrometry EPA Method 6010B', SOP LM-MI-6010B, Rev 9, February 13, 2007; also, SOP LM-MP-3050 (digestion)	Definitive	Metals, except mercury	ICP-AES	STL	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
S-5	'Mercury (Cold Vapor Technique) SW-846 Method 7471A', SOP LM-HG-7471A, Rev 11, January 31, 2007	Definitive	Mercury	Mercury auto-analyzer	STL	N
S-6	'Polychlorinated Biphenyls (PCBs) by GC/ECD SW-846 Method 8082, SOP LM-GC-8082', Rev 7, July 27, 2006; also, SOPs LM-OP-3541, LM-OP-3660B, and LM-OP-Cleanup	Definitive	Total PCB in soil and sediment	GC/ECD	STL	See Note[3]
S-7	'Total Organic Carbon in Soils and Sediment, Lloyd-Kahn Method', SOP LM-WC-TOC LK, Rev 10, February 13, 2007	Definitive	TOC in soil and sediment	Differential thermal conductivity detector	STL	N
S-8	'Particle-Size Analysis of Soils ASTM D422-63', SOP LM-SL-D422, Rev 5, August 8, 2005	Definitive	Particle-size classes in soil	Scale	STL	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
S-9	Polychlorinated Biphenyls (PCBs) by GC/ECD SW-846 Method 8082, SOP LM-GC-8082, Rev 7, July 27, 2006; also, SOPs LM-OP-3510 and LM-OP-Cleanup (Sulfuric Acid Cleanup)	Definitive	Aroclor PCB in water	GC/ECD	STL	See Note[3]
S-10	'EPA 415.1/SW-846 9060, Total Organic Carbon (Combustion)', SOP LM-WC-415.1/9060, Rev 10, November 20, 2006	Definitive	TOC in water	Catalytic combustion chamber with infrared detector	STL	N
S-11	'Determination of Total Suspended Solids (TSS) Method 160.2', SOP LM-WC-160.2, Rev 10, September 28, 2006	Definitive	TSS in water	Scale	STL	N
S-12	'Determination of Total Dissolved Solids (TDS) Method 160.1', SOP LM-WC-160.1, Rev 10, September 28, 2006	Definitive	TDS in water	Scale	STL	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
SRI/FS						
S-13	'Polychlorinated Biphenyls (PCBs) by GC/ECD SW-846 Method 8082, SOP LM-GC-8082', Rev 7, July 27, 2006; also, SOPs LM-OP-3541, LM-OP-3660B, and LM-OP-Cleanup	Definitive	Total PCB in soil and sediment	GC/ECD	STL	See Note[3]
S-14	'Extract Cleanup Procedure SW-836 Methods', SOP LM-OP-CLEANUP, Rev 7, November 6, 2006; includes SW-846 Methods 3620B, 3630C, 3600C, 3660B and 3665A	Definitive	Extract cleanup for all matrices	NA	STL	N
S-15	'Determination of Pesticides by GC/ECD CLP SOW SOM01.1', SOP LM-GC-SOM01.1-PEST, Rev 1, December 18, 2006	Definitive	Pesticides in sediment	GC/ECD	STL	N
S-16	'SOM01.1 Analytical Method for Semivolatiles EPA CLP SOM01.1', SOP LM-MS-SOM01.1-SVOA, Rev 1, January 22, 2007	Definitive	SVOCs	GC/MS	STL	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
S-17	'Analytical Method for Low/Medium Volatiles CLP SOW SOM01.0/SOM01.1', SOP LM-MV-SOM01-VOA, Rev 1, January 22, 2007	Definitive	Volatile organic compounds in sediment	GC/MS	STL	N
S-18	'CLP SOW ILM05.3/ICP-AES', SOP LM-MI-ILM05.3-ICP-AES, Rev 1, July 27, 2006	Definitive	TAL metals	ICP-AES	STL	N
S-19	'Acid Volatile Sulfide & Simultaneously Extracted Metals (AVS/SEM)', SOP LM-WC-AVS/SEM, Rev 8, November 20, 2006; also, SOP LM-MI-6010B and LM-HG-7470 for CVAA Mercury	Definitive	SEM in sediments	Titration for AVS, and ICP-AES and CVAA for SEM	STL	N
S-20	'Total Organic Carbon in Soils and Sediment, Lloyd-Kahn Method', SOP LM-WC-TOC LK, Rev 10, February 13, 2007	Definitive	TOC in soil and sediment	Differential thermal conductivity detector	STL	N
S-21	'Particle-Size Analysis of Soils ASTM D422-63', SOP LM-SL-D422, Rev 5, August 8, 2005	Definitive	Particle-size classes in soil	Scale	STL	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
S-22	Polychlorinated Biphenyls (PCBs) by GC/ECD SW-846 Method 8082, SOP LM-GC-8082, Rev 7, July 27, 2006; also, SOPs LM-OP-3510 and LM-OP-Cleanup (Sulfuric Acid Cleanup)	Definitive	Total PCB in water	GC/ECD	STL	See Note[3]
S-23	'Determination of Total Suspended Solids (TSS) Method 160.2', SOP LM-WC-160.2, Rev 10, September 28, 2006	Definitive	TSS in water	Scale	STL	N
F-7	FSP Appendix G — Fish Sampling Procedures	Definitive	Weight and length of collected fish	Measuring board or ruler, and top-loading electronic and suspended-weight spring balances	ARCADIS BBL Field Crew	N
S-24	'Polychlorinated Biphenyls (PCBs) by GC/ECD SW-846 Method 8082', SOP LM-GC-8082, Rev 7, July 27, 2006; also, SOPs LM-OP-3540, LM-OP-Cleanup, and LM-OP-GPC	Definitive	Total PCB in fish tissue	GC/ECD	STL	See Note[3]

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
S-25	'Gel Permeation Cleanup SW-846 3640', SOP LM-OP-GPC, Rev 3, May 20, 2005	Definitive	Extract cleanup of tissues for PCB analysis	HPLC	STL	N
S-26	'Percent Lipid Determination', SOP LM-OP-Lipids, Rev 6, January 3, 2007	Definitive	Lipid weight of tissue	Scale	STL	N
S-27	'Mercury in Nonpotable Water (Cold Vapor Technique)', LM-HG-7470, Rev 12, September 18, 2006	Definitive	AVS and SEM in sediments	Mercury auto-analyzer	STL	N

Notes:
[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).
[2]Specify the appropriate reference letter or number from the Field Sampling SOP References table (Worksheet #21).
[3]See Attachment 1-3 (memo and SOP dated June 26, 2006 from Jim Madison, STL, 'Kalamazoo Project PCB Procedure').

QAPP Worksheet #24 Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
TCRA						
Combined water quality meter	See F-1	See F-1	See F-1	See F-1	Analyst	F-1
Differential photometer (used in PCB Immunoassay)	Single concentration standard; 2 vials of same standard placed in instrument.	Every batch of samples.	A valid test is indicated when the magnitude of the displayed number for the 2 vials of same standard is 0.3.	If number is greater than 0.3, the instrument is outside QC limits and troubleshooting should be conducted to find problem	Analyst	F-6
GC with dual ECD, auto sampler and ChromPerfect data system with Report Write Plus PCB software	Six-point calibration of Aroclors 1016 and 1260 mixture in concentration range that brackets linear range of detector. Using the PCB Calculator Program, calibrate each Aroclor by identifying seven most prominent peaks for that particular Aroclor. All other Aroclors are quantitated based on one-point standard calibration.	After initial calibration, a check standard of Aroclor 1016/1260 mixture is analyzed every 12-hour shift with the continuing calibration standard, which is analyzed after every 20 samples or end of sequence.	Initial calibration RSD for 1016/1260 ≤ 20% or linear regression >0.99.	If routine maintenance does not return the instrument performance to meet the QC requirements, a new calibration must be performed.	Analyst	K-1 and K-2
	Continuing calibration — before sample analysis, one standard (midpoint).	1016/1260 mixture is analyzed every 12-hour shift with the continuing calibration standard, which is analyzed after every 20 samples or end of sequence.	Check standard or continuing calibration standard must not exceed a percent difference of ±15.			

QAPP Worksheet #24 Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
Analytical Balance	See SOP.	Daily.	NA.	Inspect system, correct problem, rerun calibration and affected samples.	Analyst	K-1 thru K-3,K-5,K-6,S-1 thru S-8, S-11thru S-13, S-15 thru S-20, S-22, S-23, S-25, and S-26
Spectrophotometer at 880 nm	Construct a calibration curve of absorbance vs concentration (4 standards).	Before sample analysis when check standard is outside confidence limits. Also, after major instrument maintenance.	Analyze check standard after every analytical batch. Check standard is outside 95 percent confidence limit indicated by manufacturer	Fix problem and recalibrate.		K-4
GC/MS for 8260	Instrument performance check (tune).	Prior to initial and continuing calibration.	As per method.	Retune instrument.	Analyst	S-1
	Initial calibration — prior to sample analysis, a minimum of 5 concentration levels for all compounds.	Prior to sample analysis 5 points for all compounds. Yearly.	Each compound Relative Response Factor (RRF) RSD ≤ 15% for all compounds or linear $r^2 \geq 0.99$ and RRF≥0.05.	Inspect system, correct problem, rerun calibration and affected samples if RSD > 50% or linear $r^2 \geq 0.99$.		
	Continuing calibration — before sample analysis, 1 standard (midpoint).	Before sample analysis and every 12 hours.	Each compound percent difference ≤ 20% for all compounds or linear and Response Factor (RF) ≥0.05.	Inspect system, correct problem, rerun calibration and affected samples if %D> 80%.		

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/MS for 8270	Instrument performance check (tune).	Prior to initial and continuing calibration.	As per method.	Retune instrument.	Analyst	S-2
	Initial calibration — prior to sample analysis, a minimum of 5 concentration levels for all compounds.	Prior to sample analysis, 5 points for all compounds. Yearly.	Each compound RRF RSD ≤ 15% for all compounds or linear $r^2 \geq 0.99$ and RRF≥0.05.	Inspect system, correct problem, rerun calibration and affected samples if RSD > 50% or linear $r^2 \geq 0.99$.		
	Continuing calibration — before sample analysis, 1 standard.	Before sample analysis and every 12 hours.	Each compound percent difference (%D) ≤ 20% for all compounds or linear and RF≥0.05.	Inspect system, correct problem, rerun calibration and affected samples if %D> 80%.		
GC/ECD for 8081	Initial calibration — prior to sample analysis, a minimum of 5 concentration levels for all compounds, 1 standard.	Prior to initial and continuing calibration.	Each compound Calibration (CF)≤ 20% for all compounds or linear $r^2 \geq 0.99$.	Inspect system, correct problem, rerun calibration and affected samples if CF > 20% or linear $r^2 \geq 0.99$.	Analyst	S-3
	Continuing calibration — before sample analysis, 1 standard (midpoint).	Prior to sample analysis, 5 points for all compounds. Yearly.	Each compound percent difference ≤ 15%.	Inspect system, correct problem, rerun calibration and affected samples if %D> 15%.		
ICP-AES	Minimum 3 calibration standards and calibration blank, 1 standard (midpoint).	Daily/continuing.	Initial calibration — coefficient of variation ≥.995. Continuing calibration — +/- 10% difference.	Inspect system, correct problem, rerun calibration and affected samples.	Analyst	S-4

QAPP Worksheet #24 Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
Hg cold vapor	Initial calibration — 5 concentration levels. Continuing calibration — 1 standard.	Daily, or on continuing calibration failure. Continuing, every 10 samples.	Initial calibration - ≥.995 coefficient of variation. Continuing calibration — +/- 20% difference.	Inspect system, correct problem, rerun calibration and affected samples.	Analyst	S-5
GC/ECD for 8082 modified for Kalamazoo project (STL)	Five-point ICAL standards for each Aroclor. RSD≤20%. Linearity: r≥0.99.	Before sample analysis when CCVs indicate calibration is no longer valid, and after major instrument maintenance.	After every 10 samples and end of analytical sequence, analyze two CCV Aroclor standards (Aroclor 1248, plus alternating standards for Aroclors 1242, 1254 and 1660). CCV criteria: %Difference ±15%. No Initial Calibration (ICV) standard is analyzed for this project.	Reanalyze Continuing Calibration Verification (CCV) once. If still outside criteria, recalibrate and run all samples since last successful CCV. If CCV reanalysis passes, repeat CCV analysis. Restart sample analysis if two successive CCVs pass.	Analyst	S-6, S-9, S-13, S-22, S-24
GC/MS for TPH DRO	Instrument performance check (tune). Initial Calibration — prior to sample analysis, a minimum of 5 concentration levels of Diesel Standard. Continuing calibration — before sample analysis, 1 standard.	After maintenance, perform initial calibration. Before sample analysis and every 12 hours, perform continuing calibration.	RSD of initial calibration ≤ 20%. Continuing calibration % difference is ≤ 20%.	Inspect system, correct problem, rerun calibration and affected samples.	Analyst	K-5
GC/FID for TPH GRO	Instrument performance check (tune). Initial calibration — prior to sample analysis, a minimum of 5 concentration levels of Unleaded Gasoline Standard. Continuing calibration — before sample analysis, 1 standard.	After maintenance, perform initial calibration, Before sample analysis and every 24 hours, perform continuing calibration.	Correlation coefficient (r) of GRO calibration standards > 0.995. Continuing calibration % difference is ≤ 20%.			K-6

QAPP Worksheet #24 Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
Differential thermal conductivity for Kahn TOC	Initial calibration — 5 concentration levels. Continuing calibration — 1 standard.	Daily, or on continuing calibration failure. Continuing, every 10 samples.	Initial calibration — coefficient of variation ≥.995. Laboratory control sample 75-125%.	Inspect system, correct problem, rerun calibration and affected samples.	Analyst	S-20
Catalytic combustion chamber with infrared detector for water TOC	Initial calibration — 5 concentration levels. Continuing calibration — 1 standard.	Daily, or on continuing calibration failure. Continuing, every 10 samples.	Initial calibration — coefficient of variation ≥.995. Laboratory control sample 85-115%.	Inspect system, correct problem, rerun calibration and affected samples.	Analyst	S-7
Lachat QuikChem 8000 Ion Chromatograph, Zellweger/Lachat	Initial calibration — 6 concentration levels. Continuing calibration — 1 standard.	Daily, or on continuing calibration failure. Continuing, every 10 samples.	Initial calibration — coefficient of variation ≥.997. Laboratory control sample 95-105%.	Inspect system, correct problem, rerun calibration and affected samples.	Analyst	K-7
pH meter	Initial calibration with two buffers that bracket pH of samples at room temperature.	Daily	Slope reading after calibration should be between 94-102%.	If slope falls out of range of slope reading, troubleshoot and fix problem according to user's manual.	Analyst	K-8
SRI/FS						
GC/ECD for 8082 modified for Kalamazoo project (STL)	Five-point ICAL standards for each Aroclor. RSD≤20%. Linearity: r≥0.99.	Before sample analysis when CCVs indicate calibration is no longer valid, and after major instrument maintenance.	After every 10 samples and end of analytical sequence, analyze two CCV Aroclor standards (Aroclor 1248 plus alternating standards for Aroclors 1242, 1254 and 1660). CCV criteria: % difference ±15%. No ICV standard is analyzed for this project.	Reanalyze CCV once. If still outside criteria, recalibrate and run all samples since last successful CCV. If CCV reanalysis passes, repeat CCV analysis. Restart sample analysis if two successive CCVs pass.	Analyst	S-6, S-9, S-13, S-22, S-24

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 for CLP pesticides	Initial calibration — prior to sample analysis, a minimum of 5 concentration levels for all compounds, 1 standard.	Prior to initial and continuing calibration.	As per CLP SOM 1.1	As per CLP SOM 1.1	Analyst	S-15
	Continuing calibration — before sample analysis 1 standard (midpoint).	Prior to sample analysis, 5 points for all compounds. Yearly.				
GC/MS for CLP SVOCs	Instrument performance check (tune).	Prior to initial and continuing calibration.	As per CLP SOM 1.1	As per CLP SOM 1.1	Analyst	S-16
	Initial calibration — prior to sample analysis, a minimum of 5 concentration levels for all compounds.	Prior to sample analysis, 5 points for all compounds. Yearly.				
	Continuing calibration — before sample analysis, 1 standard.	Before sample analysis and every 12 hours.				
GC/MS for CLP VOCs	Instrument performance check (tune).	Prior to initial and continuing calibration.	As per CLP SOM 1.1	As per CLP SOM 1.1	Analyst	S-17
	Initial calibration — prior to sample analysis, a minimum of 5 concentration levels for all compounds.	Prior to sample analysis, 5 points for all compounds. Yearly.				
	Continuing calibration — before sample analysis, 1 standard.	Before sample analysis and every 12 hours.				

QAPP Worksheet #24 Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
ICP-AES and cold vapor for CLP metals	As per CLP ILM 5.4	As per CLP ILM 5.4	As per CLP ILM 5.4	As per CLP ILM 5.4	Analyst	S-18

Note:
[1]Specify the appropriate SOP reference numbers correspond to from the Analytical SOP s contained in Attachment 1-3.

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/MS	• replace pump oil as needed • change gas line dryers as needed • ion source cleaning and filament replacement • replace injection port liner weekly or as needed • clip column • replace GC column as needed • manual tuning • replace electron multiplier • provide that gas supply is sufficient and delivery pressure is adequate • bake out lines and column	VOCs and SVOCs	Check connections, bake out instrument, leak test	See S-1, S-2, S-17 and S-18	See S-1, S-2, S-17 and S-18	Inspect system, correct problem, rerun calibration and affected samples	Analyst	S-1, S-2, S-17 and S-18
Purge and Trap Concentrator	• replace trap as needed • decontaminate system after high concentration samples • check for system leaks • provide that gas supply is sufficient and delivery pressure is adequate	VOCs	Check connections, bake out instrument, leak test	See S-1 and S-17	See S-1 and S-17	Inspect system, correct problem, rerun calibration and affected samples	Analyst	S-1, S-1, and S-17

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 and/or GC/FID	• 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	PCBs, pesticides and TPH	Check connections, bake out instrument, leak test	See K-1, K-2, S-3, S-6, S-9, S-13, S-15, S-22, and S-24	See K-1, K-2, S-3, S-6, S-9, S-13, S-15, S-22, and S-24	Inspect system, correct problem, rerun calibration and affected samples	Analyst	K-1, K-2, S-3, S-6, S-9, S-13, S-15, S-22, and S-24
Ion Chromatograph	• change column • change guard column • change analytical column • replace eluents	Chloride and sulfate	Check connections	See K-7	See K-7	Inspect system, correct problem, rerun calibration and affected samples	Analyst	K-7
ICP	• change capillary and pump tubing • check liquid argon tank • replace and realign plasma torch • clean nebulizer and spray chamber	All metals except mercury	Check connections, replace worn equipment	See S-4 and S-19	See S-4 and S-19	Inspect system, correct problem, rerun calibration and affected samples	Analyst	See S-4 and S-19
CVAAS	• clean tubing and quartz cell as needed • clean aspirator as necessary • check level of mercury scrubber solution • replace lamps • provide that gas supply is sufficient and delivery pressures are adequate	Mercury	Check connections, replace worn equipment	See S-5 and S-19	See S-5 and S-19	Inspect system, correct problem, rerun calibration and affected samples	Analyst	See S-5 and S-19

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]
Oven	• Thermometers checked	NA	Compare to NIST-certified thermometer	Record temperature twice daily, calibrate thermometer yearly	NA	NA	Analyst	NA
Balance	• Professional Service Contract	All	NA	3 times /year	NA	NA	Service Contractor	NA
pH Meter	• change buffer fluid • clean electrodes • rinse electrodes	NA	Inspect probe for debris	Daily	See K-8	Inspect system, correct problem, rerun calibration and affected samples	Analyst	K-8

Note:
Specify the appropriate SOP reference numbers correspond to from the Analytical SOP s contained in Attachment 1-3.

QAPP Worksheet #26-1 Sample Handling System (KAR)

Sample Collection, Packaging and Shipment
Sample Collection (Personnel/Organization): Brian Loomis/ARCADIS BBL
Sample Packaging (Personnel/Organization): Brian Loomis/ARCADIS BBL
Coordination of Shipment (Personnel/Organization): Brian Loomis/ARCADIS BBL
Type of Shipment/Carrier: ARCADIS BBL delivery to KAR
Sample Receipt and Analysis
Sample Receipt (Personnel/Organization): Mike Jaeger/KAR
Sample Custody and Storage (Personnel/Organization): Mike Jaeger/KAR
Sample Preparation (Personnel/Organization): Mike Jaeger/KAR
Sample Determinative Analysis (Personnel/Organization): Mike Jaeger/KAR
Sample Archiving
Field Sample Storage (no. of days from sample collection): 30 days from submittal of final report
Sample Extract/Digestate Storage (no. of days from extraction/digestion): 60 days (PCBs by DRO and GRO procedure)
Biological Sample Storage (no. of days from sample collection): N/A
Sample Disposal
Personnel/Organization: Mike Jaeger/KAR
Number of Days from Analysis: 60-day minimum from submittal of final report.

QAPP Worksheet #26-2 Sample Handling System (STL)

Sample Collection, Packaging and Shipment
Sample Collection (Personnel/Organization): Brian Loomis/ARCADIS BBL
Sample Packaging (Personnel/Organization): Brian Loomis/ARCADIS BBL
Coordination of Shipment (Personnel/Organization): Brian Loomis/ARCADIS BBL
Type of Shipment/Carrier: Federal Express
Sample Receipt and Analysis
Sample Receipt (Personnel/Organization): Jim Madison/STL
Sample Custody and Storage (Personnel/Organization): Jim Madison/STL
Sample Preparation (Personnel/Organization): Jim Madison/STL
Sample Determinative Analysis (Personnel/Organization): Jim Madison/STL
Sample Archiving
Field Sample Storage (no. of days from sample collection): 60 days from submittal of final report.
Sample Extract/Digestate Storage (no. of days from extraction/digestion): 60 days
Biological Sample Storage (no. of days from sample collection): Frozen until extraction; then follow above storage time for extract analysis
Sample Disposal
Personnel/Organization: Jim Madison/STL
Number of Days from Analysis: 60 days from submittal of final results.

QAPP Worksheet #27 Sample Custody Requirements

Sample Handling and Custody Requirements

Field and laboratory personnel will, at all times, be aware of the need to maintain all samples, whether in the field or in the laboratory, under strict chain of custody and in a manner to retain physical properties and chemical composition. This Worksheet details sample handling and custody requirements from collection to ultimate disposal.

Sample Handling (Sample Packaging, Shipping Containers and Sample Shipment, Sample Custody)

Sample packaging and shipment procedures are designed so that the samples will arrive at the laboratory, with the chain-of-custody, intact.

Samples will be packaged for shipment as outlined below:

- Securely affix the sample label to the container with clear packing tape.
- Check the cap on the sample container to confirm that it is properly sealed.
- Wrap the sample container cap with clear packing tape to prevent the label from becoming loose.
- Complete the chain-of-custody form with the required sampling information and confirm that the recorded information matches the sample labels. NOTE: If the designated sampler relinquishes the samples to other sampling or field personnel for packing or other purposes, the sampler will complete the chain-of-custody prior to this transfer. The appropriate personnel will sign and date the chain-of-custody form to document the sample custody transfer.
- Using duct tape, secure the outside drain plug at the bottom of the cooler.
- Wrap sample containers in bubble wrap or other cushioning material.
- Place 1 to 2 inches of cushioning material at the bottom of the cooler.
- Place the sealed sample containers into the cooler.
- Place ice in plastic bags and seal. Place loosely in the cooler.
- Fill the remaining space in the cooler with cushioning material.

QAPP Worksheet #27 Sample Custody Requirements

Sample Handling (Sample Packaging, Shipping Containers and Sample Shipment, Sample Custody) (Cont'd.)

- Place chain-of-custody forms in a plastic bag and seal. Tape the forms to the inside of the cooler lid.
- Close the lid of the cooler, lock and secure with duct tape.
- Wrap strapping tape around both ends of the cooler at least twice.
- Mark the cooler on the outside with the shipping address and return address, affix "Fragile" labels and draw (or affix) arrows indicating "this side up." Cover the labels with clear plastic tape.
- Place a signed custody seal over the sample cooler lid.

Samples will be packaged by the field personnel and transported as low-concentration environmental samples. Samples collected for VOCs analysis using EnCore® sample containers must be shipped on the day they are collected. Other samples will be hand delivered or delivered by an express carrier within 48 hours of the time of collection. Shipments will be accompanied by the chain-of-custody form identifying the contents. The original form will accompany the shipment; copies will be retained by the sampler for the sampling office records. If the samples are sent by common carrier, a bill of lading will be used. Receipts or bills of lading will be retained as part of the permanent project documentation. Commercial carriers are not required to sign off on the chain-of-custody form as long as the forms are sealed inside the sample cooler, and the custody seals remain intact.

Sample custody seals and packing materials for filled sample containers will be provided by the analytical laboratory. The filled, labeled and sealed containers will be placed in a cooler on ice and carefully packed to eliminate the possibility of container breakage.

Additional procedures for packing, handling and shipping environmental samples are presented in the Work Plan.

Field Custody Procedures

The objective of field sample custody is to protect samples from tampering from the time of sample collection through time of transport to the analytical laboratory. Persons will have custody of samples when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

Field custody documentation consists of both field logbooks and field chain-of-custody forms.

Field logbooks will provide the means of recording the data collecting activities that are performed. As such, entries will be described in as much detail as possible so that persons going to the site could reconstruct a particular situation without reliance on memory.

QAPP Worksheet #27 Sample Custody Requirements

Field Custody Procedures (Cont'd.)

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in a secure location when not in use. Each logbook will be identified by the project-specific document number. The title page of each logbook will contain the following:

- person to whom the logbook is assigned
- logbook number
- project name
- project start date
- end date

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather conditions, names of all sampling team members present, level of personal protection being used and signature of the person making the entry will be provided. The names of visitors to the site and field sampling or investigation team personnel, as well as the purpose of their visit, will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. Entries will be made in ink, with no erasures. If an incorrect entry is made, the information will be crossed out with one strike mark. Whenever a sample is collected or a measurement is made, a detailed description of the location of the station will be recorded. The number of the photographs taken, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in Section 8. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

QAPP Worksheet #27 Sample Custody Requirements

Sample Labels

Preprinted sample labels will be affixed to sample bottles prior to delivery at the sampling site. The following information is required on each sample label:

- project name
- date collected
- time collected
- location
- sampler
- analysis to be performed
- preservative
- sample number

Chain of Custody Record

Completed chain-of-custody forms will be required for all samples to be analyzed. Chain-of-custody forms will be initiated by the sampling crew in the field. The chain-of-custody forms will contain the unique sample identification number, sample date and time, sample description, sample type, preservation (if any) and analyses required. The original chain-of-custody form will accompany the samples to the laboratory. Copies of the chain-of-custody will be made prior to shipment (or multiple copy forms will be used) for field documentation. The chain-of-custody forms will remain with the samples at all times. The samples and signed chain-of-custody forms will remain in the possession of the sampling crew until the samples are delivered to the express carrier (e.g., Federal Express), hand delivered to a mobile or permanent laboratory, or placed in secure storage.

Sample labels will be completed for each sample using waterproof ink. The labels will include the information listed in the section above. The completed sample labels will be affixed to each sample bottle and covered with clear tape.

Whenever samples are split with a government agency or other party, a separate chain-of-custody will be prepared for those samples and marked to identify the party with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.

QAPP Worksheet #27 Sample Custody Requirements

Laboratory Custody Procedures
Upon sample receipt, laboratory personnel will be responsible for sample custody. The original field chain-of-custody form will accompany all samples requiring laboratory analysis. The laboratory will use chain-of-custody guidelines described in the USEPA guidance documents. Samples will be kept secured in the laboratory until all stages of analysis are complete. All laboratory personnel having samples in their custody will be responsible for documenting and maintaining sample integrity. Immediately upon sample receipt, the laboratory sample custodian will verify the integrity of the cooler seal, open the cooler and compare the contents against the field chain-of-custody. If a sample container is missing, a sample container is received broken, the sample is in an inappropriate container, or the sample has not been preserved by appropriate means, ARCADIS BBL will be notified. The laboratory sample custodian will be responsible for logging the samples in, assigning a unique laboratory identification number to each sample, labeling the sample bottle with the laboratory identification number, and moving the sample to an appropriate storage location to await analysis. The project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposition will be recorded in the laboratory tracking system. Relevant custody documentation will be placed in the project file.

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

Matrix	Water	**Analytical Method/ SOP Reference**	K-1	**No. of Sample Locations**	Numerous	
Analytical Group	PCBs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-1	**Analytical Organization**	KAR			
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 20 field samples of similar matrix	RPD < 50%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 50%
Surrogates	2 per sample	%R lab-generated limits	Reanalysis or reextraction/ reanalysis of sample	Lab personnel	Accuracy/bias	%R lab-generated limits
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reextraction/ reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample/ laboratory control* sample duplicate	1 per analytical batch	%R lab-generated limits	Reanalysis or re-extraction/reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
MS/MSD	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
MS/MSD and LCS/LCSD	1/batch	RPD lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Precision	RPD lab-generated limits

***Notes**:*
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-2 QC Samples — PCBs (Soil and Sediment) (TCRA)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	K-2	**No. of Sample Locations**	Numerous	
Analytical Group	PCBs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	KAR			
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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 100%
Surrogates	2 per sample	%R lab-generated limits	Reanalysis or reextraction/ reanalysis of sample	Lab personnel	Accuracy/bias	%R lab-generated limits
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reextraction/ reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample/ laboratory control* sample duplicate	1 per analytical batch	%R lab-generated limits	Reanalysis or reextraction/ reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
MS/MSD	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
MS/MSD and LCS/LCSD	1/batch	RPD lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Precision	RPD lab-generated limits

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-3 QC Samples — TSS and TDS (Water) (TCRA/SRI)

Matrix	Water	**Analytical Method/ SOP Reference**	K-3, S-11, S-12, and S-23	**No. of Sample Locations**	Numerous	
Analytical Group	TSS and TDS	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-1, F-10	**Analytical Organization**	KAR and STL			
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 20 field samples of similar matrix	RPD < 50%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 50%
Method blanks	1 per analytical batch	< RL	Reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample	1 per analytical batch	%R lab-generated limits	Reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
Laboratory duplicate	1 per 20 field samples of similar matrix	RPD lab-generated limits	Qualify data as needed	Lab personnel	Precision — overall	RPD lab-generated

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, laboratory duplicate, and LCS/LCSD.

QAPP Worksheet #28-4 QC Samples — Total Phosphorus (Water) (TCRA)

Matrix	Water	**Analytical Method/ SOP Reference**	K-4	**No. of Sample Locations**	Numerous	
Analytical Group	Total Phosphorus	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-1	**Analytical Organization**	KAR			
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 20 field samples of similar matrix	RPD < 50%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 50%
Method blanks	1 per analytical batch	< RL	Reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample	1 per analytical batch	%R lab-generated limits	Reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
MS	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
Laboratory duplicate	1 per 20 field samples of similar matrix	RPD lab-generated limits	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD lab-generated

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-5 QC Samples — VOCs (Soil and Sediment) (8260)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-1	**No. of Sample Locations**	Numerous	
Analytical Group	VOCs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 100%
Surrogates	3 per sample	%R lab-generated limits	Reanalyze sample	Lab personnel	Accuracy/bias	%R lab-generated limits
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	< RL
Trip blanks	1 per cooler containing VOC samples	< RL	Qualify data as needed	Lab / ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample/ laboratory control* sample duplicate	1 per analytical batch	%R lab-generated limits	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias	%R lab-generated limits
LCS/LCSD*	1/batch	%R lab-generated limits	Qualify data as needed or reanalyze batch	Lab personnel	Precision	RPD or lab-generated limits
Instrument check: BFB	1/calibration	% Relative abundance	Reanalyze batch	Lab personnel	Accuracy/bias	% Relative abundance

QAPP Worksheet #28-5 QC Samples — VOCs (Soil and Sediment) (8260)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-1	**No. of Sample Locations**	Numerous	
Analytical Group	VOCs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Internal standard	At least three/sample	Area response & retention times	Reanalyze sample	Lab personnel	Precision	Area response & retention times
MS	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
MSD	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
MS/MSD and LCS/LCSD	1/batch	RPD lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Precision	RPD lab-generated limits

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-6 QC Samples — SVOCs (Soil and Sediment) (8270)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-2	**No. of Sample Locations**	Numerous	
Analytical Group	SVOCs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 100%
Surrogates	8 per sample	%R lab-generated limits	Reanalyze sample	Lab personnel	Accuracy/bias	%R lab-generated limits
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
LCS/LCSD*	1 per analytical batch	%R lab-generated limits	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias	%R lab-generated limits
LCS/LCSD*	1/batch	%R lab-generated limits	Qualify data as needed or reanalyze batch	Lab personnel	Precision	RPD or lab-generated limits
Instrument check: bromofluorobenzene	1/calibration	% Relative abundance	Reanalyze batch	Lab personnel	Accuracy/bias	% Relative abundance
Internal standard	At least 6/sample	Area response & retention times	Reanalyze sample	Lab personnel	Precision	Area response & retention times
MS	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits

QAPP Worksheet #28-6 QC Samples — SVOCs (Soil and Sediment) (8270)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-2	**No. of Sample Locations**	Numerous	
Analytical Group	SVOCs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
MSD	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
MS/MSD and LCS/LCSD	1/batch	RPD lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Precision	RPD lab-generated limits

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-7 QC Samples — Pesticides (Soil and Sediment) (8081)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-3	**No. of Sample Locations**	Numerous	
Analytical Group	Pesticides	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 100%
Surrogates	2 per sample	%R lab-generated limits	Reanalysis or re-extraction/reanalysis of sample	Lab personnel	Accuracy/bias	%R lab-generated limits
Method blanks	1 per analytical batch	< RL	Qualify data as needed or re-extraction/reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
LCS/LCSD*	1 per analytical batch	%R lab-generated limits	Reanalysis or re-extraction/reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
MS/MSD	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS personnel	Accuracy/bias	%R lab-generated limits
MS/MSD and LCS/LCSD	1/batch	RPD lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Precision	RPD lab-generated limits

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-8 QC Samples — PCBs (SRI)

Matrix	Water	**Analytical Method/ SOP Reference**	S-9 and S-22	**No. of Sample Locations**	Numerous	
Analytical Group	PCBs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-1 and F-10	**Analytical Organization**	STL Labs			
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 20 field samples of similar matrix	RPD < 50%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 50%
Surrogates	2 per sample	%R lab-generated limits	Reanalysis or re-extraction/reanalysis of sample	Lab personnel	Accuracy/bias	%R lab-generated limits
Method blanks	1 per analytical batch	< RL	Qualify data as needed or re-extraction/reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample/laboratory control* sample duplicate	1 per analytical batch	%R lab-generated limits	Reanalysis or re-extraction/reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
MS/MSD	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
MS/MSD and LCS/LCSD	1/batch	RPD lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Precision	RPD lab-generated limits

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-9 QC Samples — PCBs (Soil, Sediment, Biota) (TCRA/SRI)

Matrix	Soil, Sediment, Biota	**Analytical Method/ SOP Reference**	S-6, S-13, S-24	**No. of Sample Locations**	Numerous	
Analytical Group	PCBs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			

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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 100%
Surrogates	2 per sample	%R lab-generated limits	Reanalysis or re-extraction/ reanalysis of sample	Lab personnel	Accuracy/bias	%R lab-generated limits
Method blanks	1 per analytical batch	< RL	Qualify data as needed or re-extraction/reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
LCS/LCSD	1 per analytical batch	%R lab-generated limits	Reanalysis or re-extraction/reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
MS/MSD	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
MS/MSD and LCS/LCSD	1/batch	RPD lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Precision	RPD lab-generated limits

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-10 QC Samples — Total Organic Carbon (Water) (TCRA)

Matrix	Water	**Analytical Method/ SOP Reference**	S-10	**No. of Sample Locations**	Numerous	
Analytical Group	TOC	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-1 and F10	**Analytical Organization**	STL			
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 20 field samples of similar matrix	RPD < 50%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 50%
Method blanks	1 per analytical batch	< RL	Reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample	1 per analytical batch	%R lab-generated limits	Reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
MS	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
Laboratory quadruplicate analysis	1 per 20 field samples of similar matrix	%D lab-generated limits	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD lab-generated

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-11 QC Samples — Total Organic Carbon

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-20	**No. of Sample Locations**	Numerous	
Analytical Group	TOC	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL Labs			
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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 100%
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample	1 per analytical batch	%R lab-generated limits	Reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
MS	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
Laboratory quadruplicate analysis	1 per 20 field samples of similar matrix	%D lab-generated limits	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD lab-generated

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-12 QC Samples — Chloride (Water)

Matrix	Water	**Analytical Method/ SOP Reference**	K-7	**No. of Sample Locations**	Numerous	
Analytical Group	Chloride	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-10	**Analytical Organization**	KAR			
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 20 field samples of similar matrix	RPD < 50%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 50%
Method blanks	1 per analytical batch	< Reporting limit (RL)	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample	1 per analytical batch	%R lab-generated limits	Reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
Matrix spike (MS)	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
Laboratory duplicate	1 per 20 field samples of similar matrix	RPD lab-generated limits	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD lab-generated limits

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-13 QC Samples — Sulfate (Water)

Matrix	Water	**Analytical Method/ SOP Reference**	K-7	**No. of Sample Locations**	Numerous	
Analytical Group	Sulfate	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-10	**Analytical Organization**	KAR			
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 20 field samples of similar matrix	RPD < 50%	Qualify data as needed	ARCADIS BBL personnel	Precision - overall	RPD < 50%
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample	1 per analytical batch	%R lab-generated limits	Reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
MS	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
Laboratory duplicate	1 per 20 field samples of similar matrix	RPD lab-generated limits	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD lab-generated limits

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-14 QC Samples — Total Alkalinity (Water)

Matrix	Water	**Analytical Method/ SOP Reference**	K-8	**No. of Sample Locations**	Numerous	
Analytical Group	Total Alkalinity	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-10	**Analytical Organization**	KAR Labs			
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 20 field samples of similar matrix	RPD < 50%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 50%
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample	1 per analytical batch	%R lab-generated limits	Reanalysis of batch	Lab personnel	Accuracy/bias	%R lab-generated limits
MS	1/batch	%R lab-generated limits	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
Laboratory duplicate	1 per 20 field samples of similar matrix	RPD lab-generated limits	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD lab-generated limits

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-15 QC Samples — Metals (Water) (6000)

Matrix	Water	**Analytical Method/ SOP Reference**	S-4	**No. of Sample Locations**	Numerous	
Analytical Group	Metals	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-10	**Analytical Organization**	STL			

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 20 field samples of similar matrix	RPD < 50%	Qualify data as needed	Lab personnel and/or ARCADIS BBL personnel	Precision — overall	RPD < 50%
Method blanks	Numerous	< RL	Qualify data as needed or reanalysis of batch	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Calibration verification standards	Numerous	%R (90-110%)	Reanalysis of batch	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias contamination	%R (90-110%)
Interference check sample (A and AB)	2/run	Certain metals (%R (80-120%)	Qualify data as needed or reanalysis of batch	Lab personnel and/or ARCADIS BBL personnel	Precision — lab	Certain metals %R (80-120%)
MS	1/batch	%R lab-generated limits	Qualify data as needed	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
Laboratory duplicate	1 per 20 field samples of similar matrix	RPD lab-generated limits	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD lab-generated
LCS	1/batch	%R lab-generated limits	Qualify data as needed or reanalysis of batch	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits

QAPP Worksheet #28-15 QC Samples — Metals (Water) (6000)

Matrix	Water	**Analytical Method/ SOP Reference**	S-4	**No. of Sample Locations**	Numerous	
Analytical Group	Metals	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-10	**Analytical Organization**	STL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Post digestion spike	1/batch	%R (75-125%)	Qualify data as needed	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias	%R (75-125%)
Serial dilution	1/batch	%D < 10%	Qualify data as needed	Lab personnel and/or ARCADIS BBL personnel	Precision	%D < 10%

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-16 QC Samples — Metals (Soil and Sediment) (6000/7000)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-4, S-5, and S-19	**No. of Sample Locations**	Numerous	
Analytical Group	Metals	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 100%
Method blanks	Numerous	< RL	Qualify data as needed or reanalysis of batch	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Calibration verification standards	Numerous	%R (90-110%)	Reanalysis of batch	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias contamination	%R (90-110%)
Interference check sample (A and AB)	2/run	Certain metals %R (80-120%)	Qualify data as needed or reanalysis of batch	Lab personnel and/or ARCADIS BBL personnel	Precision — lab	Certain metals %R (80-120%)
MS	1/batch	%R lab-generated limits	Qualify data as needed	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits
Laboratory duplicate	1 per 20 field samples of similar matrix	RPD lab-generated limits	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD lab-generated
LCS	1/batch	%R lab-generated limits	Qualify data as needed or reanalysis of batch	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias	%R lab-generated limits

QAPP Worksheet #28-16 QC Samples — Metals (Soil and Sediment) (6000/7000)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-4, S-5, and S-19	**No. of Sample Locations**	Numerous	
Analytical Group	Metals	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Post-digestion spike	1/batch	%R (75-125%)	Qualify data as needed	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias	%R (75-125%)
Serial dilution	1/batch	%D < 10%	Qualify data as needed	Lab personnel and/or ARCADIS BBL personnel	Precision	%D < 10%

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 1-3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-17 QC Samples — VOCs (Soil and Sediment) (SOM)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-17	**No. of Sample Locations**	Numerous	
Analytical Group	VOCs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 100%
Surrogates	14 per sample	%R limits listed in USEPA CLP-SOM Form 2	If more than 3 surrogates< or > limits[1] or one surrogate <10% the sample is reanalyze sample	Lab personnel	Accuracy/bias	%R limits listed in USEPA CLP-SOM Form 2
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	< RL
Instrument blanks	1 per following a sample or sample dilution that exceeds the calibration range	< RL	Qualify data as needed or reanalyze associated samples	Lab personnel	Accuracy/bias contamination	< RL
Storage blanks	1 per SDG	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	< RL
Trip blanks	1 per cooler containing VOC samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL

QAPP Worksheet #28-17 QC Samples — VOCs (Soil and Sediment) (SOM)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-17	**No. of Sample Locations**	Numerous	
Analytical Group	VOCs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Laboratory control sample/laboratory control* sample duplicate	1 per analytical batch	%R limits listed in USEPA CLP-SOM Form 3	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias	%R limits listed in USEPA CLP-SOM Form 3
LCS/LCSD	1/batch	%R lab-generated limits	Qualify data as needed or reanalyze batch	Lab personnel	Precision	%R lab-generated limits
Instrument check: BFB	1/calibration	% Relative abundance	Reanalyze batch	Lab personnel	Accuracy/bias	% Relative abundance
Internal standard	3 per sample	%R limits listed in USEPA CLP-SOM Form 8	Reanalyze sample	Lab personnel	Precision	%R limits listed in USEPA CLP-SOM Form 8
MS	1/batch	%R limits listed in USEPA CLP-SOM Form 3	Qualify data as needed or reanalyze associated samples	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R limits listed in USEPA CLP-SOM Form 3
MSD	1/batch	%R limits listed in USEPA CLP-SOM Form 3	Qualify data as needed or reanalyze associated samples	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R limits listed in USEPA CLP-SOM Form 3
MS/MSD and LCS/LCSD	1/batch	RPD limits listed in USEPA CLP-SOM Form 3	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Precision	%R limits listed in USEPA CLP-SOM Form 3

Notes:
[1] The surrogate recovery limits associated with 1,4-dioxane –d_8 are advisory.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-18 QC Samples — SVOCs (Soil and Sediment) (SOM)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-16	**No. of Sample Locations**	Numerous	
Analytical Group	SVOCs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	ARCADIS BBL personnel	Precision — overall	RPD < 100%
Surrogates	16 per sample	%R limits listed in USEPA CLP-SOM Form 2	If more than 4 surrogates< or > limits none may be <10% the sample is re-extracted and reanalyze sample	Lab personnel	Accuracy/bias	%R limits listed in USEPA CLP-SOM Form 2
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	< RL
Instrument blanks	1 per following a sample or sample dilution that exceeds the calibration range	< RL	Qualify data as needed or reanalyze associated samples	Lab personnel	Accuracy/bias contamination	< RL
Storage blanks	1 per SDG	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	< RL
Trip blanks	1 per cooler containing VOC samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL

QAPP Worksheet #28-18 QC Samples — SVOCs (Soil and Sediment) (SOM)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-16	**No. of Sample Locations**	Numerous	
Analytical Group	SVOCs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
LCS/LCSD*	1 per analytical batch	%R limits listed in USEPA CLP-SOM Form 3	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias	%R limits listed in USEPA CLP-SOM Form 3
LCS/LCSD*	1/batch	%R lab-generated limits	Qualify data as needed or reanalyze batch	Lab personnel	Precision	%R lab-generated limits
Instrument check: BFB	1/calibration	% Relative abundance	Reanalyze batch	Lab personnel	Accuracy/bias	% Relative abundance
Internal standard	6 per sample	%R limits listed in USEPA CLP-SOM Form 8	Reanalyze sample	Lab personnel	Precision	%R limits listed in USEPA CLP-SOM Form 8
MS	1/batch	%R limits listed in USEPA CLP-SOM Form 3	Qualify data as needed or reanalyze associated samples	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R limits listed in USEPA CLP-SOM Form 3
MSD	1/batch	%R limits listed in USEPA CLP-SOM Form 3	Qualify data as needed or reanalyze associated samples	Lab and/or ARCADIS BBL personnel	Accuracy/bias	%R limits listed in USEPA CLP-SOM Form 3
MS/MSD and LCS/LCSD	1/batch	RPD limits listed in USEPA CLP-SOM Form 3	Qualify data as needed	Lab and/or ARCADIS BBL personnel	Precision	%R limits listed in USEPA CLP-SOM Form 3

Note:
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-19 QC Samples — Pesticides (Soil and Sediment) (SOM)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-3	**No. of Sample Locations**	Numerous	
Analytical Group	Pesticides	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	ARCADIS BBL personnel	Precision - overall	RPD < 100%
Surrogates	2 per sample	% Recovery (%R) limits listed in USEPA CLP-SOM Form 2	If more than 1 surrogate < or > limits or one surrogate <10% of the sample, reanalyze sample	Lab personnel	Accuracy/bias	If more than 1 surrogate < or > limits or one surrogate <10% of the sample, re-analyze sample
Method blanks	1 per analytical batch	< Reporting limit (RL)	Qualify data as needed or re-extraction/ reanalysis of batch	Lab personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Laboratory control sample/laboratory control* sample duplicate	1 per analytical batch	%R limits listed in USEPA CLP-SOM Form 3	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias	%R limits listed in USEPA CLP-SOM Form 3
Matrix spike (MS)/ matrix spike duplicate (MSD)	1/batch	RPD limits listed in USEPA CLP-SOM Form 3	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias	%R limits listed in USEPA CLP-SOM Form 3
MS/MSD	1/batch	%R limits listed in USEPA CLP-SOM Form 3	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias	%R limits listed in USEPA CLP-SOM Form 3

Note:
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS/MSD, and LCS/LCSD.

QAPP Worksheet #28-20 QC Samples — PCBs (Immunoassay) (Soil and Sediment) (TCRA)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	F-6	**No. of Sample Locations**	Numerous	
Analytical Group	PCBs (immunoassay)	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	ARCADIS BBL			
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 20 field samples of similar matrix	RPD < 100%	Reanalysis of samples	ARCADIS BBL personnel	Precision — overall	RPD < 100%

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.

QAPP Worksheet #28-21 QC Samples — Lipids

Matrix	Biota	**Analytical Method/ SOP Reference**	S-26	**No. of Sample Locations**	Numerous	
Analytical Group	PCBs	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-8	**Analytical Organization**	STL Labs			
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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	ARCADIS personnel	Precision — overall	RPD < 100%
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	< RL
Laboratory duplicate	1 per 20 field samples of similar matrix	<25%	Qualify data as needed or reanalyze batch	Lab personnel	Precision — overall	<25%

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, and laboratory duplicates.

QAPP Worksheet #28-22 QC Samples — Temperature, pH, Turbidity, Conductivity, DO and Specific Conductance (Water)

Matrix	Water	**Analytical Method/ SOP Reference**	F-1 and F-10	**No. of Sample Locations**	Numerous	
Analytical Group	Temperature, pH, Turbidity, Conductivity, DO and Specific Conductance	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-1 and F-10	**Analytical Organization**	ARCADIS BBL			
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 20 field samples of similar matrix	RPD < 50%	Reanalyze sample or recalibrate and reanalyze sample	ARCADIS BBL personnel	Precision — overall	RPD < 50%

Notes:
Laboratory generated limits referred to throughout Worksheet 28 are presented in Attachment 3 and will be updated annually when the laboratories revise the internal control limits for parameters included in this QAPP.
*LCS/LCSD used when MS/MSD not client-supplied.

QAPP Worksheet #28-23 QC Samples — Metals (Soil and Sediment) (ILM)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-18 and S-27	**No. of Sample Locations**	Numerous	
Analytical Group	Metals	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
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 20 field samples of similar matrix	RPD < 100%	Qualify data as needed	Lab personnel and/or ARCADIS BBL personnel	Precision - overall	RPD < 100%
Method blanks	Numerous	< Reporting limit (RL)	Qualify data as needed or reanalyze batch	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	ARCADIS BBL personnel	Accuracy/bias contamination	< RL
Calibration verification standards	Numerous	%R (90-110%) except for Hg %R (80-120%)	Reanalyze batch	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias contamination	%R (90-110%) except for Hg %R (80-120%)
Interference check sample (A and AB)	2/run	Certain metals %R (80-120%)	Qualify data as needed or reanalyze batch	Lab personnel and/or ARCADIS BBL personnel	Precision - lab	Certain metals %R (80-120%)
Matrix spike	1/batch	75% to 125%	Qualify data as needed	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias	75% to 125%
Laboratory duplicate	1 per 20 field samples of similar matrix	<20%	Qualify data as needed	ARCADIS BBL personnel	Precision - overall	<20%
Laboratory control sample (LCS)	1/batch	< limits reported on Form 7	Qualify data as needed or reanalyze batch	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias	< limits reported on Form 7

QAPP Worksheet #28-23 QC Samples — Metals (Soil and Sediment) (ILM)

Matrix	Soil and Sediment	**Analytical Method/ SOP Reference**	S-18 and S-27	**No. of Sample Locations**	Numerous	
Analytical Group	Metals	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	ARCADIS BBL			
Sampling SOP	F-4 and F-5	**Analytical Organization**	STL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Post-digestion spike	1/batch	%R (75-125%)	Qualify data as needed	Lab personnel and/or ARCADIS BBL personnel	Accuracy/bias	%R (75-125%)
Serial dilution	1/batch	% Difference (%D) < 10%	Qualify data as needed	Lab personnel and/or ARCADIS BBL personnel	Precision	%D < 10%

Note:
An analytical batch is defined as no more than 20 analytical sample including field samples, blanks, MS, laboratory duplicates and LCS/LCSD.

QAPP Worksheet #29 Project Documents and Records

Sample Collection Documents and Records	On-site Analysis Documents and Records[1]	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 Samples, Standards, QC Checks and QC Samples - Data Package Completeness Checklists - Sample Disposal Records - Extraction/Cleanup Records - Raw Data (stored on disk or CD-R) - Analytical Reports to ARCADIS BBL	- Data Validation Checklists - Data Quality Assessments	- FSP - HSP - QAPP - Data Management Plan - Pilot Study Summary Reports - Risk Assessments - Routine Monitoring Reports - Record of Decision documents - Proposed Plans - Feasibility Studies - Long-Term Monitoring Reports

Note:
Information generated for the TCRA monitoring will be temporarily stored on site as appropriate during TCRA construction activities and long-term in ARCADIS BBL's Brighton, Michigan office. Information from the SRI/FS will be stored in ARCADIS BBL's Brighton, Michigan office.

QAPP Worksheet #30 Analytical Services

Matrix	Analytical Group	Concentration Level	Analytical SOP	Data Package Turnaround Time (calendar days)	Laboratory/Organization (name and address, contact person and telephone number)	Backup Laboratory/Organization (name and address, contact person and telephone number)
Surface Water	PCBs, TSS, total phosphorus	All	See Worksheet #23	10 days for EDD and PDF of Level 2, Data Packages	KAR Laboratories, Inc. 4425 Manchester Road Kalamazoo, MI 49001 269.381.9666	NA
Groundwater	Chloride, sulfate, alkalinity	All	See Worksheet #23	10 days for EDD and PDF of Level 2, and 20 days for Level 4 Data Packages	KAR Laboratories, Inc. 4425 Manchester Road Kalamazoo, MI 49001 269.381.9666	NA
Soil/sediment	TPH-DRO, TPH-GRO	All	See Worksheet #23	10 days for EDD and PDF of Level 2, and 20 days for Level 4 Data Packages	KAR Laboratories, Inc. 4425 Manchester Road Kalamazoo, MI 49001 269.381.9666	NA
Soil/sediment	PCBs	All	See Worksheet #23	24 hours for EDD and PDF of Level 2, and 20 days for Level 4 Data Packages	KAR Laboratories, Inc. 4425 Manchester Road Kalamazoo, MI 49001 269.381.9666	STL Burlington 30 Community Drive, Suite 11 South Burlington, VT 05403 802.660.1990
Surface water/ groundwater/ soil/ sediment/ biota	PCBs, TOC, VOCs, SVOCs, pesticides, metals, particle size, TDS, TSS, AVS/SEM, percent lipids	All	See Worksheet #23	10 days for EDD and PDF of Level 2 and 20 days for Level 4 Data Packages	STL Burlington 30 Community Drive, Suite 11 South Burlington, VT 05403 802.660.1990	NA

QAPP Worksheet #31 Planned Project Assessments

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 (CAs) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
TCRA							
Confirmation Monitoring Field Audit	twice per year	Internal	ARCADIS BBL	Construction Quality Assurance (CQA) Engineer, ARCADIS BBL	Rich Price, Field Program Manager, ARCADIS BBL	E.J. Suardini, Construction Manager, ARCADIS BBL	Rich Price, Field Program Manager, ARCADIS BBL
Laboratory Audit of KAR	As necessary based on laboratory performance	External	ARCADIS BBL	Dennis Capria, Data QA Manager, ARCADIS BBL	Mike Jaeger, Laboratory Project Manager, KAR	Linda Felcyn, Laboratory QA Manager, KAR	Steve Garbaciak, TRCA Project Coordinator, ARCADIS BBL
Laboratory Audit of STL	As necessary based on laboratory performance	External	ARCADIS BBL	Dennis Capria, Data QA Manager, ARCADIS BBL	Jim Madison, Project Manager, STL	Kirstin L. McCracken, Laboratory QA Manager, STL	Steve Garbaciak, TRCA Project Coordinator, ARCADIS BBL
Laboratory Audit	Per Laboratory Quality Assurance Program	Internal	KAR and STL	Laboratory QA Manager	Laboratory QA Manager	Laboratory Project Manager	Dennis Capria, Data QA Manager, ARCADIS BBL

QAPP Worksheet #31 Planned Project Assessments

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 (CAs) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Field Inspections	Intermittent	Internal	ARCADIS BBL	Matt Bowman, Field Program Coordinator, ARCADIS BBL	Rich Price, Field Program Manager, ARCADIS BBL	E.J. Suardini, Construction Manager, ARCADIS BBL	Rich Price, Field Program Manager, ARCADIS BBL
Project Audit	Intermittent	Internal	ARCADIS BBL	J. Paul Doody, Vice President, ARCADIS BBL	Mike Erickson, SRI Project Coordinator, ARCADIS BBL	Various, Program Managers, ARCADIS BBL	Mike Erickson, SRI Project Coordinator, ARCADIS BBL
Safety Audits	Annually and LPS Field Assessment based on > 400 field hours/ quarter	Internal	ARCADIS BBL	Corporate H&S Staff, Regional H&S Manager or Regional H&S Specialist	Rich Price, Field Program Manager, ARCADIS BBL	E.J. Suardini, Construction Manager, ARCADIS BBL	Rich Price, Field Program Manager, ARCADIS BBL

QAPP Worksheet #31 Planned Project Assessments

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 (CAs) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
SRI/FS							
Field Audit (Sediment Program)	1 per year	Internal	ARCADIS BBL	Todd Merrell, Field Team Leader, ARCADIS BBL	Mike Scoville, SRI Field Program Coordinator, ARCADIS BBL	Brian Loomis, Field Program Manager, ARCADIS BBL	Mike Scoville, SRI Field Program Coordinator, ARCADIS BBL
Laboratory Audit of KAR Laboratory	As necessary based on laboratory performance	External	ARCADIS BBL	Dennis Capria, Data QA Manager, ARCADIS BBL	Mike Scoville, SRI Field Program Coordinator, ARCADIS BBL	Linda Felcyn Laboratory QA Manager, KAR Labs	Steve Garbaciak, TRCA Project Coordinator, ARCADIS BBL
Laboratory Audit of STL	As necessary, based on laboratory performance	External	ARCADIS BBL	Dennis Capria, Data QA Manager, ARCADIS BBL	Mike Scoville, SRI Field Program Coordinator, ARCADIS BBL	Jim Madison, Laboratory Project Manager, STL	Mike Scoville, SRI Field Program Coordinator, ARCADIS BBL
Laboratory Audit	Per laboratory Quality Assurance Program	Internal	ARCADIS BBL	Dennis Capria, Data QA Manager, ARCADIS BBL	Mike Scoville, SRI Field Program Coordinator, ARCADIS BBL	Jim Madison, Laboratory Project Manager, STL	Mike Scoville, SRI Field Program Coordinator, ARCADIS BBL

QAPP Worksheet #31 Planned Project Assessments

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 (CAs) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Field Inspections	Intermittent	Internal	ARCADIS BBL	Todd Merrell, Field Team Leader, ARCADIS BBL	Mike Scoville, SRI Field Program Coordinator, ARCADIS BBL	Brian Loomis, Field Program Manager, ARCADIS BBL	Mike Scoville, SRI Field Program Coordinator, ARCADIS BBL
Project Audit	Intermittent	Internal	ARCADIS BBL	J. Paul Doody, Vice President, ARCADIS BBL	Mike Erickson, SRI Project Coordinator, ARCADIS BBL	Various, Program Managers, ARCADIS BBL	Mike Erickson, SRI Project Coordinator, ARCADIS BBL
Safety Audits	Annually and LPS Field Assessment based on > 400 field hours/ quarter	Internal	ARCADIS BBL	Corporate H&S Staff, Regional H&S Manager or Regional H&S Specialist	Mike Scoville, SRI Field Program Coordinator, ARCADIS BBL	Brian Loomis, Field Program Manager, ARCADIS BBL	Mike Scoville, SRI Field Program Coordinator, ARCADIS BBL
Groundwater Monitoring Program Field Audit	1 per year	Internal	ARCADIS BBL	Senior Hydrogeologist, ARCADIS BBL	Brian Loomis, Field Program Manager, ARCADIS BBL	Brian Loomis, Field Program Manager, ARCADIS BBL	Senior Hydrogeologist, ARCADIS BBL

QAPP Worksheet #32 Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Field Sampling Technical Systems Audit	Written Audit Report	Mike Erickson (SRI/FS Project Coordinator) and Steve Garbaciak (Plainwell TCRA Project Coordinator)	72 hours after audit	Letter	Mike Scoville (SRI Field Program Coordinator) and Brian Loomis (Field Program Manager)	48 hours after notification
Contract Laboratory Technical Audit (external)	Written Audit Report	Mike Jaeger (KAR Project Manager), Jim Madison (STL Project Manager), Mike Erickson (SRI/FS Project Coordinator) and Steve Garbaciak (Plainwell TCRA Project Coordinator) and Dennis Capria (Data QA Manager)	1 week after audit	Letter	Linda Felcyn (KAR Laboratory QA Manager) and Kirstin L. McCracken (STL Laboratory QA Manager)	48 hours after notification
Field Inspection	Memorandum	Brian Loomis (Field Program Manager)	2 days	Memorandum	Mike Scoville (SRI Field Program Coordinator)	48 hours after notification
Contract Laboratory Technical Audit (internal)	Memorandum	Mike Jaeger (KAR Laboratory Project Manager), Jim Madison (STL Laboratory Project Manager), and Dennis Capria (Data QA Manager)	2 days	Memorandum	Linda Felcyn (KAR Lab QA Manager) and Kirstin L. McCracken (STL Lab QA Manager)	48 hours after notification

QAPP Worksheet #33 QA Management Reports

Type of Report	Frequency (e.g., daily, weekly monthly, quarterly, annually)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Field Sampling Technical Systems Audit Report	1/year	NA	Mike Scoville (SRI Field Program Coordinator) and Brian Loomis (Field Program Manager)	Mike Erickson (SRI/FS Project Coordinator) and Steve Garbaciak (Plainwell TCRA Project Coordinator)
Contract Laboratory Technical Audit Report	As necessary during project	NA	Dennis Capria (Data QA Manager)	Mike Erickson (SRI/FS Project Coordinator) and Steve Garbaciak (Plainwell TCRA Project Coordinator)
Data Validation Reports	As specified in the Data Assessment section based on intended use of the data and required percentage to meet project DQOs	As generated throughout project	Dennis Capria (Data QA Manager)	Mike Erickson (SRI/FS Project Coordinator) and Steve Garbaciak (Plainwell TCRA Project Coordinator)
Data Quality Summary Reports	As appropriate for data use and as required for report completion	As generated throughout project	Dennis Capria (Data QA Manager)	Mike Erickson (SRI/FS Project Coordinator) and Steve Garbaciak (Plainwell TCRA Project Coordinator)

QAPP Worksheet #34 Verification (Step I) Process

Verification Input	Description	Internal/ External	Responsible for Verification (name, organization)
Chain-of-custody and shipping forms	Chain-of-custody forms and shipping documentation will be reviewed by laboratory upon receipt of samples for verification against the sample coolers they represent. Chain-of-custody form will be signed by all parties that had custody of samples, with the exception of commercial carriers.	External	KAR and STL
Field notes and sampling logs	All field notes and sampling logs will be reviewed internally and placed in the project file.	Internal	ARCADIS BBL
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	Linda Felcyn, KAR and Kirstin L. McCracken, STL
Laboratory data	All final data packages will be verified for content upon receipt.	External	Dennis Capria, ARCADIS BBL

QAPP Worksheet #35 Validation (Steps IIa and IIb) Process

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
Step IIa	Sampling methods and procedures	Establish that required sampling methods were used and that any deviations were noted. Provide that the sampling procedures and field measurements met performance criteria and that any deviations were documented.	Brian Loomis, ARCADIS BBL
Step IIa	Analytical method and procedures	Establish that required analytical methods were used and that any deviations were noted. The laboratory will provide that QC samples met performance criteria and that any deviations were documented in the report narrative.	Linda Felcyn, KAR, Kirstin L. McCracken ,STL, Dennis Capria, ARCADIS BBL
Step IIa Modified	Analytical method and procedures	Review associated blanks for potential contamination and verify that all preparations and analyses have been performed within applicable holding times.	Dennis Capria, ARCADIS BBL
Step IIb	Documentation of QAPP QC sample results	Establish that all QAPP-required QC samples were collected and analyzed.	Dennis Capria, ARCADIS BBL
Step IIb	Project quantitation limits	Determine that the project quantitation limits were achieved, as outlined in the QAPP.	Dennis Capria, ARCADIS BBL
Step IIb	Performance criteria	Evaluate QC data associated with 25% of the samples designated for intended uses stated in Worksheet #36 against project-specific performance criteria in the QAPP, laboratory Quality Assurance Manual and control criteria.	Dennis Capria and Mike Scoville ARCADIS BBL
Step IIb	Validation Report	Summarize data verification and validation components included in the Performance Review. Include qualified data and explanation of all qualifiers.	Dennis Capria, ARCADIS BBL

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

Step IIa/IIb	Matrix	Analytical Group	Data Purpose	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
TCRA						
IIa and IIb	Aqueous/soil/ sediment	PCBs, VOCs, SVOCs, pesticides	Contaminant delineation, risk assessment, confirmation of remediation	Low, medium, high	USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 1999 and USEPA Region II SOPs that apply to SW-846 methods; method criteria; laboratory control limits; QAPP criteria; and professional judgment	Dennis Capria (Data QA Manager, ARCADIS BBL)
IIa and IIb	Aqueous/soil/ sediment	Metals, general chemistry parameters	Contaminant delineation, risk assessment, confirmation of remediation	Low, medium, high	USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, October 2002 and USEPA Region II SOPs that apply to SW-846 methods; laboratory control limits; method criteria; QAPP criteria; and professional judgment	Dennis Capria (Data QA Manager, ARCADIS BBL)
SRI/FS						
IIa and IIb	Aqueous/soil/ sediment/biota	PCBs	Contaminant delineation, risk assessment, confirmation of remediation	Low, medium, high	USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 1999 and USEPA Region II SOPs that apply to SW-846 methods; method criteria; laboratory control limits; QAPP criteria; and professional judgment	Dennis Capria (Data QA Manager, ARCADIS BBL)
IIa and IIb	Aqueous/soil/ sediment/biota	VOCs, SVOCs and pesticides	Contaminant delineation, risk assessment, confirmation of remediation	Low, medium, high	USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, Finial Draft January 2005; method criteria; QAPP criteria; and professional judgment	Dennis Capria (Data QA Manager, ARCADIS BBL)

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

Step IIa/IIb	Matrix	Analytical Group	Data Purpose	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Aqueous/soil/ sediment/biota	Metals, general chemistry parameters	Contaminant delineation, risk assessment, confirmation of remediation	Low, medium, high	USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, October 2004 and USEPA Region II SOPs that apply to SW-846 methods; laboratory control limits; method criteria; QAPP criteria; and professional judgment	Dennis Capria (Data QA Manager, ARCADIS BBL)

QAPP Worksheet #37 Usability Assessment

The Data Usability Assessment will be performed by ARCADIS BBL for data associated with delineation, risk assessment or confirmation of remedial achievement. Documentation generated during the usability assessment will consist of data validation checklists with a brief summary of overall data usability.

The Data Usability Assessment process involves data verification and data validation. Data verification is the process by which laboratory results are checked to provide that the proper quality control steps were performed and key items have met QC objectives (both analytical and contractual). The key items checked in an ARCADIS BBL data verification include:

- sample collection, handling and analysis procedures
- field sampling, handling and analysis activities will be documented (e.g., QC signatures in field logs, QC checklist)
- sampling, handling, on-site analytical and off-site laboratory data will be verified internally at the data generator level
- laboratory data (e.g., laboratory-qualified data)
- sampling, on-site analytical and off-site laboratory data
- data package deliverable completeness
- review of case narrative
- present all analytical results
- QC sample data summaries
- applicable raw data

All required data deliverables must be present in the data package in order to proceed to the next step of data validation.

QAPP Worksheet #37 Usability Assessment

Data validation entails a review of the sample collection, handling, field analysis and QC data, and the raw data to verify that the laboratory was operating within required limits; analytical results were correctly transcribed from the instrument read-outs; and which (if any) environmental samples were related to out-of-control QC samples. The objective of data validation is to identify any questionable or invalid laboratory measurements.

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

DQIs are generally defined in terms of 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.

QAPP Worksheet #37 Usability Assessment

Comparability

Comparability is the degree of confidence with which one data set can be compared to 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 this QAPP, established QA/QC procedures and 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 to 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.

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 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.

QAPP Worksheet #37 Usability Assessment

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

QAPP Worksheet #37 Usability Assessment

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. The current MDLs for the analytical laboratories are presented in Worksheet #15.

Data Validation and Usability

ARCADIS BBL will validate all data generated using the most recent versions of the USEPA's National Functional Guidelines (USEPA 1999; 2002) and USEPA Region II SOPs TCRA associated with SW-846 analysis and USEPA's National Functional Guidelines (USEPA 1999; 2004; 2005) and USEPA Region II SOPs SRI/FS associated with SW-846 and CLP analysis for data validation available at the time of project initiation, where appropriate. These procedures and criteria may be modified, as necessary, to address project-specific and method-specific criteria, control limits and procedures. Data validation will consist of data screening, checking, reviewing, editing and interpretation to document analytical data quality and to determine whether the quality is sufficient to meet the DQOs.

The data validator will verify that reduction of laboratory measurements and laboratory reporting of analytical parameters is in accordance with the procedures specified for each analytical method and/or as specified in this QAPP. Any deviations from the analytical method or any special reporting requirements apart from those specified in this QAPP will be detailed on COC forms.

Upon receipt of laboratory data, the following procedures will be executed by the data validator:

- Evaluate completeness of data package.
- Verify that field COC forms were completed and that samples were handled properly.
- Verify that holding times were met for each parameter. Holding time exceedances, should they occur, will be documented. Data for all samples exceeding holding time requirements will be flagged as either estimated or rejected. The decision as to which qualifier is more appropriate will be made on a case-by-case basis.
- Verify that parameters were analyzed according to the methods specified.

QAPP Worksheet #37 Usability Assessment

- Review QA/QC data (i.e., confirm that duplicates, blanks and spikes were analyzed on the required number of samples, as specified in the method and verify that duplicate and MS recoveries are acceptable).

- Investigate anomalies identified during review. When anomalies are identified, they will be discussed with the Project Manager and/or Laboratory Manager, as appropriate.

- If data appear suspect, investigate the specific data of concern. Calculations will be traced back to raw data. If calculations do not agree, the cause will be determined and corrected.

Deficiencies discovered as a result of the data review, as well as the corrective actions implemented in response, will be documented and submitted in the form of a written report addressing the following topics, as applicable to each method:

- assessment of the data package

- description of any protocol deviations

- failures to reconcile reported and/or raw data

- assessment of any compromised data

- overall appraisal of the analytical data

- table of site name, sample quantities, matrix and fractions analyzed

It should be noted that qualified results do not necessarily invalidate data. The goal to produce the best possible data does not necessarily mean that data must be produced without QC qualifiers. Qualified data can provide useful information.

QAPP Worksheet #37 Usability Assessment

During the review process, laboratory qualified and unqualified data are verified against the supporting documentation. Based on this evaluation, qualifier codes may be added, deleted, or modified by the data reviewer. Results will be qualified with the following codes in accordance with the USEPA National Functional Guidelines:

Concentration (C) qualifiers:

U The analyte/compound was analyzed for but not detected. The associated value is the compound quantitation limit.

Inorganics:

B The reported value was obtained from a reading less than the required reporting limit (RL), but greater than or equal to the instrument.

Quantitation (Q) qualifiers:

Inorganics:

E The reported value is estimated due to the presence of interference.

N Spiked sample recovery not within control limits.

* Duplicate analysis not within control limits.

Organics:

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

B The compound has been found in the sample as well as its associated blank; its presence in the sample may be suspect.

N The analysis indicates the presence of a compound for which there is presumptive evidence to make a tentative identification.

QAPP Worksheet #37 Usability Assessment

P The lower of the two values is reported when the percent difference between the results of two GC columns is greater than 25 percent.

E The compound was quantitated above the calibration range.

D Concentration is based on a diluted sample analysis.

C Identification confirmed by gas chromatography/mass spectrometry (GC/MS).

Validation qualifiers:

U The analyte/compound was analyzed for, but not detected. The associated value is the compound quantitation limit.

UJ The compound was not detected above the reported sample quantitation limit. 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.

JN The analysis indicates the presence of a compound for which there is presumptive evidence to make a tentative identification. The associated numerical value is an estimated concentration only.

R The sample results are rejected.

Two facts will be noted to all data users. First, the "R" flag means that the associated value is unusable. In other words, due to significant QC problems, the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data, but any value potentially contains error.

Resolution of any issues regarding laboratory performance or deliverables will be handled between the laboratory and the data validator. Suggestions for reanalysis may be made by the QAC at this point.

QAPP Worksheet #37 Usability Assessment

Validation Reports

The data validation reports will identify all deficiencies and the potential impact on the results. The Validation and Database Project Team will amend qualifiers generated during the validation process to the database. The validation checklists and the database will be the primary location of all applicable data qualifiers. Qualifiers will not be applied to the hard copy analytical reports.

Field Data Review

Field data are generated from in-field measurement, which may include a geophysical survey, well development, groundwater sampling and surface-water sampling. The quality objective for the in-field measurement activities is to obtain accurate measurements of sample characteristics, including pH, conductivity, temperature, turbidity, dissolved oxygen and/or redox potential, using appropriate equipment. Data are recorded in field logbooks or on field sampling sheets and calibration logs. Calibration logs will be reviewed with other field documentation to identify any potential impacts to data quality and usability. Field logbooks are reviewed as part of the QC inspections.

Reconciliation with Data Usability Requirements

The data results will be examined to determine the performance that was achieved for each data usability criterion. The performance will then be compared with the project objectives and DQOs. Deviations from objectives will be noted. Additional action may be warranted when performance does not meet performance objectives for critical data. Options for corrective action relating to incomplete information, questionable results, or inconsistent data may include any or all of the following:

- retrieval of missing information
- request for additional explanation or clarification
- reanalysis of sample from extract (when appropriate)
- recalculation or reinterpretation of results by the laboratory

These actions may improve the data quality, reduce uncertainty and eliminate the need to qualify or reject data.

QAPP Worksheet #37 Usability Assessment

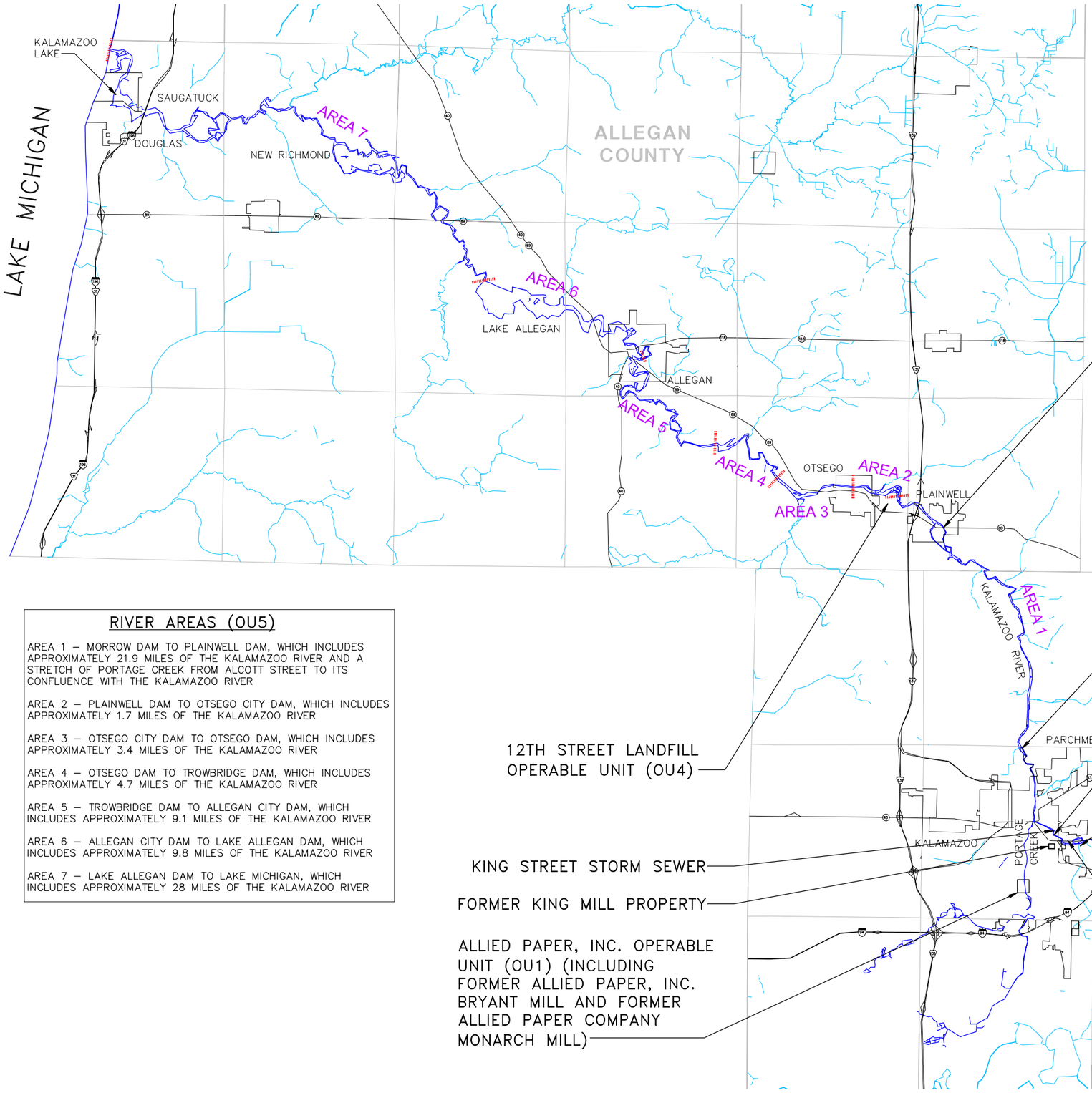
If these actions do not improve the data quality to an acceptable level, the following additional actions may be taken:

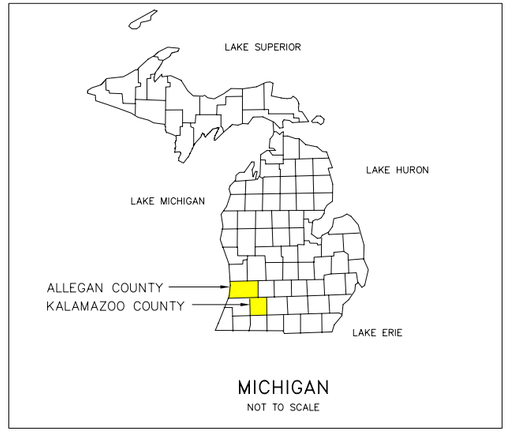
- extrapolation of missing data from existing data points
- use of historical data
- evaluation of the critical/noncritical nature of the sample

If the data gap cannot be resolved by these actions, the data bias and potential for false negatives and positives can be evaluated. If the resultant uncertainty level is unacceptable, the following action must be taken:

- additional sample collection and analysis

FIGURES





PLAINWELL MILL PROPERTY OPERABLE UNIT (OU7)

PARCHMENT MILL PROPERTY

KING HIGHWAY LANDFILL OPERABLE UNIT (OU3)

WILLOW BOULEVARD/A-SITE OPERABLE UNIT (OU2)

12TH STREET LANDFILL OPERABLE UNIT (OU4)

KING STREET STORM SEWER

FORMER KING MILL PROPERTY

ALLIED PAPER, INC. OPERABLE UNIT (OU1) (INCLUDING FORMER ALLIED PAPER, INC. BRYANT MILL AND FORMER ALLIED PAPER COMPANY MONARCH MILL)

GEORGIA-PACIFIC CORPORATION KALAMAZOO MILL AND HAWTHORNE MILL PROPERTIES (SEE NOTE 2)

NOTES:

1. ALLEGAN AND KALAMAZOO COUNTY MAPPING OBTAINED FROM MICHIGAN RESOURCE INFORMATION SYSTEM.
2. OU6 IS CURRENTLY A RELATIVELY SMALL PART OF THE PARCEL COMPRISING BOTH MILLS.

LEGEND:

LIMITS OF RIVER AREAS WITHIN OU5

RIVER AREAS (OU5)

AREA 1 – MORROW DAM TO PLAINWELL DAM, WHICH INCLUDES APPROXIMATELY 21.9 MILES OF THE KALAMAZOO RIVER AND A STRETCH OF PORTAGE CREEK FROM ALCOTT STREET TO ITS CONFLUENCE WITH THE KALAMAZOO RIVER

AREA 2 – PLAINWELL DAM TO OTSEGO CITY DAM, WHICH INCLUDES APPROXIMATELY 1.7 MILES OF THE KALAMAZOO RIVER

AREA 3 – OTSEGO CITY DAM TO OTSEGO DAM, WHICH INCLUDES APPROXIMATELY 3.4 MILES OF THE KALAMAZOO RIVER

AREA 4 – OTSEGO DAM TO TROWBRIDGE DAM, WHICH INCLUDES APPROXIMATELY 4.7 MILES OF THE KALAMAZOO RIVER

AREA 5 – TROWBRIDGE DAM TO ALLEGAN CITY DAM, WHICH INCLUDES APPROXIMATELY 9.1 MILES OF THE KALAMAZOO RIVER

AREA 6 – ALLEGAN CITY DAM TO LAKE ALLEGAN DAM, WHICH INCLUDES APPROXIMATELY 9.8 MILES OF THE KALAMAZOO RIVER

AREA 7 – LAKE ALLEGAN DAM TO LAKE MICHIGAN, WHICH INCLUDES APPROXIMATELY 28 MILES OF THE KALAMAZOO RIVER

0 10,000 FEET 20,000 FEET
GRAPHIC SCALE

KALAMAZOO RIVER STUDY GROUP
ALLIED PAPER, INC./PORTAGE CREEK/
KALAMAZOO RIVER SUPERFUND SITE
MULTI-AREA QUALITY ASSURANCE PROJECT PLAN

AREAS OF THE SITE

ARCADIS BBL
Infrastructure, environment, facilities

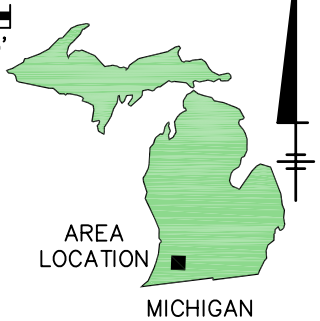
FIGURE
1-1

[SYR-85-LEAD] SYR-85-GMS KEW RCA LAYER: ON=*; OFF=REF
G:\CAD\ACTIVE\-DWG\ACT\64524500\BOARDS\64524B05.DWG SAVED:4/9/2007 2:59 PM LAYOUT:1 PAGESETUP:---- PENTABLE:PLTFULL.CTB PRINTED:4/9/2007 3:00 PM BY:RALLEN
PROJECTNAME:----
XREFS: IMAGES:

REFERENCE: USGS QUADS., 7.5 MIN. SERIES, DRG TOPOGRAPHIC MAP - ALLEGAN COUNTY, MICHIGAN.

0 2000' 4000'

Approximate Scale: 1" = 2000'



KALAMAZOO RIVER STUDY GROUP
ALLIED PAPER, INC./PORTAGE CREEK/
KALAMAZOO RIVER SUPERFUND SITE
QUALITY ASSURANCE PROJECT PLAN

SITE LOCATION MAP (TCRA)

ARCADIS BBL
Infrastructure, environment, facilities

FIGURE
1-2

[SYR-85-KLS] SYR-85-KLS KLS KFS LAYER: ON=*, OFF=*REF*
G:\CAD\ACTIVE\-DWG\ACT\64530670\TCRA-C\64530N01.dwg SAVED: 4/4/2007 10:55 AM LAYOUT: SiteLocMap PAGESETUP: ---- PENTABLE: PLTFULL.CTB PRINTED: 4/4/2007 11:01 AM BY: KSTINSON
PROJECTNAME: ----
XREFS: IMAGES: 64530X04.tif

ARCADIS BBL

ATTACHMENTS

ARCADIS BBL

Attachment 1-1

Chain of Custody

ARCADIS
Infrastructure, environment, facilities

ID#:

CHAIN OF CUSTODY & LABORATORY ANALYSIS REQUEST FORM

Page ___ of ___

Lab Work Order #

Send Results to:

Contact & Company Name:	Telephone:
Address:	Fax:
City State Zip	E-mail Address:
Project Name/Location (City, State):	Project #:
Sampler's Printed Name:	Sampler's Signature:

Preservative							
Filtered (✓)							
# of Containers							
Container Information							

PARAMETER ANALYSIS & METHOD

Keys

Preservation Key:
A. H_2SO_4
B. HCL
C. HNO_3
D. NaOH
E. None
F. Other: _____
G. Other: _____
H. Other: _____

Container Information Key:
1. 40 ml Vial
2. 1 L Amber
3. 250 ml Plastic
4. 500 ml Plastic
5. Encore
6. 2 oz. Glass
7. 4 oz. Glass
8. 8 oz. Glass
9. Other: _____
10. Other: _____

Matrix Key:
SO - Soil
W - Water
T - Tissue
SE - Sediment
SL - Sludge
A - Air
NL - NAPL/Oil
SW - Sample Wipe
Other: _____

Sample ID	Collection Date	Collection Time	Type (✓) Comp	Type (✓) Grab	Matrix								REMARKS

Special Instructions/Comments:

☐ **Special QA/QC Instructions(✓):**

Laboratory Information and Receipt		Relinquished By	Received By	Relinquished By	Laboratory Received By
Lab Name:	Cooler Custody Seal (✓)	Printed Name:	Printed Name:	Printed Name:	Printed Name:
☐ Cooler packed with ice (✓)	☐ Intact ☐ Not Intact	Signature:	Signature:	Signature:	Signature:
Specify Turnaround Requirements:	Sample Receipt:	Firm:	Firm/Courier:	Firm/Courier:	Firm:
Shipping Tracking #:	Condition/Cooler Temp: _____	Date/Time:	Date/Time:	Date/Time:	Date/Time:

Distribution: **WHITE – Laboratory returns with results** **YELLOW – Lab copy** **PINK – Retained by BBL**

ARCADIS BBL

Attachment 1-2

Laboratory Certifications

State of New Jersey
Department of Environmental Protection

Certifies That

Severn Trent Laboratories, Inc. / STL Burlington

Laboratory Certification ID#: VT972

having duly met the requirements of the

Regulations Governing The Certification Of
Laboratories And Environmental Measurements N.J.A.C. 7:18 et. seq.

and

having been found compliant with the standard approved by the

National Environmental Laboratory Accreditation Conference

is hereby approved as a

Nationally Accredited Environmental Laboratory

to perform the analyses as indicated on the Annual Certified Parameter List
which must accompany this certificate to be valid

Expiration Date June 30, 2007

Joseph F. Aiello, Chief
Office of Quality Assurance

NJDEP is a NELAP Recognized Accrediting Authority

THIS CERTIFICATE IS TO BE CONSPICUOUSLY DISPLAYED AT THE LABORATORY WITH THE ANNUAL CERTIFIED PARAMETER LIST IN A LOCATION ON THE PREMISES VISIBLE TO THE PUBLIC

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: CAP01 -- Atmos. Inorg. Parameters, Non-Metals

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CAP01.00005	AE	Thermal Conductivity	[EPA 3C]	Carbon Dioxide
Certified	Yes	NJ	CAP01.00068	AE	Thermal Conductivity	[EPA 3C]	Methane
Certified	Yes	NJ	CAP01.00070	AE	Thermal Conductivity	[EPA 3C]	Nitrogen
Certified	Yes	NJ	CAP01.00100	AE	Thermal Conductivity	[EPA 3C]	Oxygen

Category: CAP03 -- Atmospheric Organic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CAP03.00015	AE	FID	[EPA 25C]	Non-Methane Organic Compounds
Certified	Yes	NJ	CAP03.00184	AE	GC/MS, Canisters	[EPA TO-15]	Acetone
Certified	Yes	NJ	CAP03.00225	AE	GC/MS, Canisters	[EPA TO-15]	Benzene
Certified	Yes	NJ	CAP03.00250	AE	GC/MS, Canisters	[EPA TO-15]	Bromodichloromethane
Certified	Yes	NJ	CAP03.00255	AE	GC/MS, Canisters	[EPA TO-15]	Bromoform
Certified	Yes	NJ	CAP03.00260	AE	GC/MS, Canisters	[EPA TO-15]	Bromomethane
Certified	Yes	NJ	CAP03.00265	AE	GC/MS, Canisters	[EPA TO-15]	Butadiene (1,3-)
Certified	Yes	NJ	CAP03.00270	AE	GC/MS, Canisters	[EPA TO-15]	Carbon disulfide
Certified	Yes	NJ	CAP03.00275	AE	GC/MS, Canisters	[EPA TO-15]	Carbon tetrachloride
Certified	Yes	NJ	CAP03.00300	AE	GC/MS, Canisters	[EPA TO-15]	Chlorobenzene
Certified	Yes	NJ	CAP03.00305	AE	GC/MS, Canisters	[EPA TO-15]	Chloroethane
Certified	Yes	NJ	CAP03.00310	AE	GC/MS, Canisters	[EPA TO-15]	Chloroform
Certified	Yes	NJ	CAP03.00315	AE	GC/MS, Canisters	[EPA TO-15]	Chloromethane
Certified	Yes	NJ	CAP03.00325	AE	GC/MS, Canisters	[EPA TO-15]	Chlorotoluene (2-)
Certified	Yes	NJ	CAP03.00335	AE	GC/MS, Canisters	[EPA TO-15]	Cyclohexane
Certified	Yes	NJ	CAP03.00342	AE	GC/MS, Canisters	[EPA TO-15]	Dibromochloromethane
Certified	Yes	NJ	CAP03.00350	AE	GC/MS, Canisters	[EPA TO-15]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	CAP03.00355	AE	GC/MS, Canisters	[EPA TO-15]	Dichlorobenzene (1,2-)
Certified	Yes	NJ	CAP03.00360	AE	GC/MS, Canisters	[EPA TO-15]	Dichlorobenzene (1,3-)
Certified	Yes	NJ	CAP03.00365	AE	GC/MS, Canisters	[EPA TO-15]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	CAP03.00368	AE	GC/MS, Canisters	[EPA TO-15]	Dichlorodifluoromethane
Certified	Yes	NJ	CAP03.00370	AE	GC/MS, Canisters	[EPA TO-15]	Dichloroethane (1,1-)

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: CAP03 -- Atmospheric Organic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CAP03.00375	AE	GC/MS, Canisters	[EPA TO-15]	Dichloroethane (1,2-)
Certified	Yes	NJ	CAP03.00380	AE	GC/MS, Canisters	[EPA TO-15]	Dichloroethene (1,1-)
Certified	Yes	NJ	CAP03.00384	AE	GC/MS, Canisters	[EPA TO-15]	Dichloroethene (cis-1,2-)
Certified	Yes	NJ	CAP03.00385	AE	GC/MS, Canisters	[EPA TO-15]	Dichloroethene (trans-1,2-)
Certified	Yes	NJ	CAP03.00395	AE	GC/MS, Canisters	[EPA TO-15]	Dichloropropane (1,2-)
Certified	Yes	NJ	CAP03.00400	AE	GC/MS, Canisters	[EPA TO-15]	Dichloropropene (cis-1,3-)
Certified	Yes	NJ	CAP03.00401	AE	GC/MS, Canisters	[EPA TO-15]	Dichloropropene (trans-1,3-)
Certified	Yes	NJ	CAP03.00405	AE	GC/MS, Canisters	[EPA TO-15]	Dichlorotetrafluoroethane (1,2-)
Certified	Yes	NJ	CAP03.00440	AE	GC/MS, Canisters	[EPA TO-15]	Dioxane (1,4-)
Certified	Yes	NJ	CAP03.00465	AE	GC/MS, Canisters	[EPA TO-15]	Ethylbenzene
Certified	Yes	NJ	CAP03.00480	AE	GC/MS, Canisters	[EPA TO-15]	Ethyltoluene (4-)
Certified	Yes	NJ	CAP03.00490	AE	GC/MS, Canisters	[EPA TO-15]	Hexachlorobutadiene (1,3-)
Certified	Yes	NJ	CAP03.00498	AE	GC/MS, Canisters	[EPA TO-15]	Hexanone (2-)
Certified	Yes	NJ	CAP03.00500	AE	GC/MS, Canisters	[EPA TO-15]	Heptane (n-)
Certified	Yes	NJ	CAP03.00505	AE	GC/MS, Canisters	[EPA TO-15]	Hexane (n-)
Certified	Yes	NJ	CAP03.00511	AE	GC/MS, Canisters	[EPA TO-15]	Isopropanol
Certified	Yes	NJ	CAP03.00525	AE	GC/MS, Canisters	[EPA TO-15]	Methyl ethyl ketone
Certified	Yes	NJ	CAP03.00535	AE	GC/MS, Canisters	[EPA TO-15]	Methyl isobutyl ketone
Certified	Yes	NJ	CAP03.00550	AE	GC/MS, Canisters	[EPA TO-15]	Methyl tert-butyl ether
Certified	Yes	NJ	CAP03.00555	AE	GC/MS, Canisters	[EPA TO-15]	Methylene chloride (Dichloromethane)
Certified	Yes	NJ	CAP03.00625	AE	GC/MS, Canisters	[EPA TO-15]	Styrene
Certified	Yes	NJ	CAP03.00635	AE	GC/MS, Canisters	[EPA TO-15]	Trichlorobenzene (1,2,4-)
Certified	Yes	NJ	CAP03.00640	AE	GC/MS, Canisters	[EPA TO-15]	Trimethylbenzene (1,3,5-)
Certified	Yes	NJ	CAP03.00645	AE	GC/MS, Canisters	[EPA TO-15]	Trimethylbenzene (1,2,4-)
Certified	Yes	NJ	CAP03.00650	AE	GC/MS, Canisters	[EPA TO-15]	Trimethylpentane (2,2,4-)
Certified	Yes	NJ	CAP03.00652	AE	GC/MS, Canisters	[EPA TO-15]	Tert-butyl alcohol
Certified	Yes	NJ	CAP03.00655	AE	GC/MS, Canisters	[EPA TO-15]	Tetrachloroethane (1,1,2,2-)
Certified	Yes	NJ	CAP03.00660	AE	GC/MS, Canisters	[EPA TO-15]	Tetrachloroethene
Certified	Yes	NJ	CAP03.00665	AE	GC/MS, Canisters	[EPA TO-15]	Toluene
Certified	Yes	NJ	CAP03.00670	AE	GC/MS, Canisters	[EPA TO-15]	Trichloroethane (1,1,1-)
Certified	Yes	NJ	CAP03.00675	AE	GC/MS, Canisters	[EPA TO-15]	Trichloroethane (1,1,2-)
Certified	Yes	NJ	CAP03.00680	AE	GC/MS, Canisters	[EPA TO-15]	Trichloroethene

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: CAP03 -- Atmospheric Organic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CAP03.00684	AE	GC/MS, Canisters	[EPA TO-15]	Trichlorofluoromethane
Certified	Yes	NJ	CAP03.00685	AE	GC/MS, Canisters	[EPA TO-15]	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
Certified	Yes	NJ	CAP03.00710	AE	GC/MS, Canisters	[EPA TO-15]	Vinyl chloride
Certified	Yes	NJ	CAP03.00715	AE	GC/MS, Canisters	[EPA TO-15]	Xylene (m-)
Certified	Yes	NJ	CAP03.00720	AE	GC/MS, Canisters	[EPA TO-15]	Xylene (o-)
Certified	Yes	NJ	CAP03.00725	AE	GC/MS, Canisters	[EPA TO-15]	Xylene (p-)
Certified	Yes	NJ	CAP03.00730	AE	GC/MS, Canisters	[EPA TO-15]	Xylenes (total)
Certified	Yes	NJ	CAP03.06150	AE	GC/MS	[EPA TO-13A]	Acenaphthene
Certified	Yes	NJ	CAP03.06160	AE	GC/MS	[EPA TO-13A]	Acenaphthylene
Certified	Yes	NJ	CAP03.06170	AE	GC/MS	[EPA TO-13A]	Anthracene
Certified	Yes	NJ	CAP03.06180	AE	GC/MS	[EPA TO-13A]	Benzo(a)anthracene
Certified	Yes	NJ	CAP03.06190	AE	GC/MS	[EPA TO-13A]	Benzo(a)pyrene
Certified	Yes	NJ	CAP03.06200	AE	GC/MS	[EPA TO-13A]	Benzo(b)fluoranthene
Certified	Yes	NJ	CAP03.06210	AE	GC/MS	[EPA TO-13A]	Benzo(k)fluoranthene
Certified	Yes	NJ	CAP03.06230	AE	GC/MS	[EPA TO-13A]	Benzo(ghi)perylene
Certified	Yes	NJ	CAP03.06240	AE	GC/MS	[EPA TO-13A]	Chrysene
Certified	Yes	NJ	CAP03.06260	AE	GC/MS	[EPA TO-13A]	Dibenzo(a,h)anthracene
Certified	Yes	NJ	CAP03.06270	AE	GC/MS	[EPA TO-13A]	Fluoranthene
Certified	Yes	NJ	CAP03.06280	AE	GC/MS	[EPA TO-13A]	Fluorene
Certified	Yes	NJ	CAP03.06290	AE	GC/MS	[EPA TO-13A]	Indeno(1,2,3-cd)pyrene
Certified	Yes	NJ	CAP03.06300	AE	GC/MS	[EPA TO-13A]	Naphthalene
Certified	Yes	NJ	CAP03.06320	AE	GC/MS	[EPA TO-13A]	Phenanthrene
Certified	Yes	NJ	CAP03.06330	AE	GC/MS	[EPA TO-13A]	Pyrene
Certified	Yes	NJ	CAP03.06430	AE	GC/MS, CANISTERS	[EPA TO-14A]	Benzene
Certified	Yes	NJ	CAP03.06450	AE	GC/MS, CANISTERS	[EPA TO-14A]	Bromomethane
Certified	Yes	NJ	CAP03.06460	AE	GC/MS, CANISTERS	[EPA TO-14A]	Carbon tetrachloride
Certified	Yes	NJ	CAP03.06470	AE	GC/MS, CANISTERS	[EPA TO-14A]	Chlorobenzene
Certified	Yes	NJ	CAP03.06480	AE	GC/MS, CANISTERS	[EPA TO-14A]	Chloroethane
Certified	Yes	NJ	CAP03.06490	AE	GC/MS, CANISTERS	[EPA TO-14A]	Chloroform
Certified	Yes	NJ	CAP03.06500	AE	GC/MS, CANISTERS	[EPA TO-14A]	Chloromethane
Certified	Yes	NJ	CAP03.06510	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	CAP03.06520	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichlorobenzene (1,2-)

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

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Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: CAP03 -- Atmospheric Organic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CAP03.06530	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichlorobenzene (1,3-)
Certified	Yes	NJ	CAP03.06540	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	CAP03.06550	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichlorodifluoromethane
Certified	Yes	NJ	CAP03.06560	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloroethane (1,1-)
Certified	Yes	NJ	CAP03.06570	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloroethane (1,2-)
Certified	Yes	NJ	CAP03.06580	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloroethene (1,1-)
Certified	Yes	NJ	CAP03.06590	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloroethene (cis-1,2-)
Certified	Yes	NJ	CAP03.06610	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloropropane (1,2-)
Certified	Yes	NJ	CAP03.06620	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloropropene (cis-1,3-)
Certified	Yes	NJ	CAP03.06630	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichloropropene (trans-1,3-)
Certified	Yes	NJ	CAP03.06640	AE	GC/MS, CANISTERS	[EPA TO-14A]	Dichlorotetrafluoroethane (1,2-)
Certified	Yes	NJ	CAP03.06650	AE	GC/MS, CANISTERS	[EPA TO-14A]	Ethylbenzene
Certified	Yes	NJ	CAP03.06660	AE	GC/MS, CANISTERS	[EPA TO-14A]	Hexachlorobutadiene (1,3-)
Certified	Yes	NJ	CAP03.06670	AE	GC/MS, CANISTERS	[EPA TO-14A]	Styrene
Certified	Yes	NJ	CAP03.06680	AE	GC/MS, CANISTERS	[EPA TO-14A]	Tetrachloroethane (1,1,2,2-)
Certified	Yes	NJ	CAP03.06690	AE	GC/MS, CANISTERS	[EPA TO-14A]	Tetrachloroethene
Certified	Yes	NJ	CAP03.06700	AE	GC/MS, CANISTERS	[EPA TO-14A]	Toluene
Certified	Yes	NJ	CAP03.06710	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trichlorobenzene (1,2,4-)
Certified	Yes	NJ	CAP03.06720	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trichloroethane (1,1,1-)
Certified	Yes	NJ	CAP03.06730	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trichloroethene
Certified	Yes	NJ	CAP03.06740	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trichlorofluoromethane
Certified	Yes	NJ	CAP03.06760	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trimethylbenzene (1,2,4-)
Certified	Yes	NJ	CAP03.06770	AE	GC/MS, CANISTERS	[EPA TO-14A]	Trimethylbenzene (1,3,5-)
Certified	Yes	NJ	CAP03.06780	AE	GC/MS, CANISTERS	[EPA TO-14A]	Vinyl chloride
Certified	Yes	NJ	CAP03.06790	AE	GC/MS, CANISTERS	[EPA TO-14A]	Xylene (o-)
Certified	Yes	NJ	CAP03.06800	AE	GC/MS, CANISTERS	[EPA TO-14A]	Xylene (m-)
Certified	Yes	NJ	CAP03.06810	AE	GC/MS, CANISTERS	[EPA TO-14A]	Xylene (p-)

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

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National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: SDW02 -- Inorganic Parameters Including Na + Ca

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW02.04000	DW	Ion Chromatography	[EPA 300.0]	Nitrate
Certified	Yes	NJ	SDW02.09000	DW	Spectrophotometric	[SM 4500-NO2 B]	Nitrite
Certified	Yes	NJ	SDW02.14000	DW	Ion Chromatography	[EPA 300.0]	Fluoride
Certified	Yes	NJ	SDW02.15200	DW	Spectrophotometric, Distill, Semi Automated	[EPA 335.4]	Cyanide
Applied	No	NJ	SDW02.19000	DW	Ion Chromatography	[EPA 300.0]	Sulfate
Dropped	No	NJ	SDW02.20000	DW	ICP	[EPA 200.7]	Sodium
Certified	Yes	NJ	SDW02.27000	DW	ICP	[EPA 200.7]	Calcium
Applied	No	NJ	SDW02.27300	DW	Hardness By Calculation	[EPA 200.7]	Total hardness
Certified	Yes	NJ	SDW02.31000	DW	Ion Chromatography	[EPA 300.0]	Chloride
Certified	Yes	NJ	SDW02.31120	DW	Ion Chromatography	[EPA 314.0]	Perchlorate
Certified	Yes	NJ	SDW02.31125	DW	LC MS/MS	[EPA 331.0]	Perchlorate
Certified	No	NJ	SDW02.36400	DW	ICP	[EPA 200.7]	Silica

Category: SDW03 -- Analyze-Immediately Inorganic Parameter

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW03.08000	DW	Electrometric	[EPA 150.1]	pH

Category: SDW04 -- Inorganic Parameters, Metals

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Dropped	No	NJ	SDW04.03000	DW	ICP	[EPA 200.7]	Aluminum
Certified	Yes	NJ	SDW04.07000	DW	ICP/MS	[EPA 200.8]	Antimony
Certified	Yes	NJ	SDW04.12000	DW	ICP/MS	[EPA 200.8]	Arsenic
Dropped	No	NJ	SDW04.16000	DW	ICP	[EPA 200.7]	Barium
Dropped	No	NJ	SDW04.20000	DW	ICP	[EPA 200.7]	Beryllium
Dropped	No	NJ	SDW04.24000	DW	ICP	[EPA 200.7]	Cadmium
Dropped	No	NJ	SDW04.28000	DW	ICP	[EPA 200.7]	Chromium
Dropped	No	NJ	SDW04.33000	DW	ICP	[EPA 200.7]	Copper

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

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208 SOUTH PARK DR
STE 1

Category: SDW04 -- Inorganic Parameters, Metals

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Dropped	No	NJ	SDW04.37000	DW	ICP	[EPA 200.7]	Iron
Certified	Yes	NJ	SDW04.40000	DW	ICP/MS	[EPA 200.8]	Lead
Dropped	No	NJ	SDW04.44000	DW	ICP	[EPA 200.7]	Manganese
Certified	Yes	NJ	SDW04.46000	DW	Manual Cold Vapor	[EPA 245.1]	Mercury
Dropped	No	NJ	SDW04.52000	DW	ICP	[EPA 200.7]	Nickel
Certified	Yes	NJ	SDW04.57000	DW	ICP/MS	[EPA 200.8]	Selenium
Dropped	No	NJ	SDW04.62000	DW	ICP	[EPA 200.7]	Silver
Certified	Yes	NJ	SDW04.65000	DW	ICP/MS	[EPA 200.8]	Thallium
Dropped	No	NJ	SDW04.67000	DW	ICP	[EPA 200.7]	Zinc

Category: SDW05 -- Organic Parameters, Chromatography

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW05.12010	DW	Solvent Extract, GC	[EPA 504.1]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	SDW05.12020	DW	Solvent Extract, GC	[EPA 504.1]	Dibromo-3-chloropropane (1,2-)
Certified	Yes	NJ	SDW05.12030	DW	Solvent Extract, GC	[EPA 504.1]	Trichloropropane (1,2,3-)

Category: SDW06 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW06.01010	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Bromoform
Certified	Yes	NJ	SDW06.01020	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chloroform
Certified	Yes	NJ	SDW06.01030	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dibromochloromethane
Certified	Yes	NJ	SDW06.01040	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Bromodichloromethane
Certified	Yes	NJ	SDW06.02010	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Benzene
Certified	Yes	NJ	SDW06.02020	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Carbon tetrachloride
Certified	Yes	NJ	SDW06.02030	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chlorobenzene
Certified	Yes	NJ	SDW06.02040	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichlorobenzene (1,2-)
Certified	Yes	NJ	SDW06.02050	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichlorobenzene (1,3-)

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Category: SDW06 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW06.02060	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	SDW06.02070	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloroethane (1,1-)
Certified	Yes	NJ	SDW06.02080	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloroethane (1,2-)
Certified	Yes	NJ	SDW06.02090	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloroethene (cis-1,2-)
Certified	Yes	NJ	SDW06.02100	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloroethene (trans-1,2-)
Certified	Yes	NJ	SDW06.02110	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methylene chloride (Dichloromethane)
Certified	Yes	NJ	SDW06.02120	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropane (1,2-)
Certified	Yes	NJ	SDW06.02130	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Ethylbenzene
Certified	Yes	NJ	SDW06.02140	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methyl tert-butyl ether
Certified	Yes	NJ	SDW06.02150	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Naphthalene
Certified	Yes	NJ	SDW06.02160	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Styrene
Certified	Yes	NJ	SDW06.02170	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tetrachloroethane (1,1,2,2-)
Certified	Yes	NJ	SDW06.02180	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tetrachloroethene
Certified	Yes	NJ	SDW06.02190	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichloroethane (1,1,1-)
Certified	Yes	NJ	SDW06.02200	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichloroethene
Certified	Yes	NJ	SDW06.02210	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Toluene
Certified	Yes	NJ	SDW06.02220	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichlorobenzene (1,2,4-)
Certified	Yes	NJ	SDW06.02230	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloroethene (1,1-)
Certified	Yes	NJ	SDW06.02240	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichloroethane (1,1,2-)
Certified	Yes	NJ	SDW06.02250	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Vinyl chloride
Certified	Yes	NJ	SDW06.02260	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Xylenes (total)
Certified	Yes	NJ	SDW06.03010	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Bromobenzene
Certified	Yes	NJ	SDW06.03020	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Bromochloromethane
Certified	Yes	NJ	SDW06.03030	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Bromomethane
Certified	Yes	NJ	SDW06.03040	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Butyl benzene (n-)
Certified	Yes	NJ	SDW06.03050	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Sec-butylbenzene
Certified	Yes	NJ	SDW06.03060	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tert-butylbenzene
Certified	Yes	NJ	SDW06.03070	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chloroethane
Certified	Yes	NJ	SDW06.03080	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chloromethane
Certified	Yes	NJ	SDW06.03090	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chlorotoluene (2-)
Certified	Yes	NJ	SDW06.03100	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chlorotoluene (4-)
Certified	Yes	NJ	SDW06.03110	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dibromo-3-chloropropane (1,2-)

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208 SOUTH PARK DR
STE 1

Category: SDW06 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW06.03120	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	SDW06.03130	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dibromomethane
Certified	Yes	NJ	SDW06.03140	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichlorodifluoromethane
Certified	Yes	NJ	SDW06.03150	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropane (1,3-)
Certified	Yes	NJ	SDW06.03160	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropane (2,2-)
Certified	Yes	NJ	SDW06.03170	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropene (1,1-)
Certified	Yes	NJ	SDW06.03180	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropene (cis-1,3-)
Certified	Yes	NJ	SDW06.03190	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropene (trans-1,3-)
Certified	Yes	NJ	SDW06.03200	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Hexachlorobutadiene (1,3-)
Certified	Yes	NJ	SDW06.03210	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Isopropylbenzene
Certified	Yes	NJ	SDW06.03220	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Isopropyltoluene (4-)
Certified	Yes	NJ	SDW06.03230	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Propylbenzene (n-)
Certified	Yes	NJ	SDW06.03240	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tetrachloroethane (1,1,1,2-)
Certified	Yes	NJ	SDW06.03250	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichlorobenzene (1,2,3-)
Certified	Yes	NJ	SDW06.03251	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichlorobenzene (1,3,5-)
Certified	Yes	NJ	SDW06.03260	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichlorofluoromethane
Certified	Yes	NJ	SDW06.03270	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trichloropropane (1,2,3-)
Certified	Yes	NJ	SDW06.03280	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trimethylbenzene (1,2,4-)
Certified	Yes	NJ	SDW06.03300	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Trimethylbenzene (1,3,5-)
Certified	Yes	NJ	SDW06.03310	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Nitrobenzene
Certified	Yes	NJ	SDW06.03410	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Acetone
Certified	Yes	NJ	SDW06.03420	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Acrylonitrile
Certified	Yes	NJ	SDW06.03430	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Allyl chloride
Certified	Yes	NJ	SDW06.03440	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Butanone (2-)
Certified	Yes	NJ	SDW06.03450	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Carbon disulfide
Certified	Yes	NJ	SDW06.03460	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chloroacetonitrile
Certified	Yes	NJ	SDW06.03470	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Chlorobutane (1-)
Certified	Yes	NJ	SDW06.03480	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloro-2-butene (trans-1,4-)
Certified	Yes	NJ	SDW06.03490	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Dichloropropanone (1,1-)
Certified	Yes	NJ	SDW06.03500	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Diethyl ether (Ethyl ether)
Certified	Yes	NJ	SDW06.03510	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Ethyl methacrylate
Certified	Yes	NJ	SDW06.03520	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Hexachloroethane

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208 SOUTH PARK DR
STE 1

Category: SDW06 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SDW06.03530	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Hexanone (2-)
Certified	Yes	NJ	SDW06.03540	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methacrylonitrile
Certified	Yes	NJ	SDW06.03550	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methyl acrylate
Certified	Yes	NJ	SDW06.03560	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methyl iodide
Certified	Yes	NJ	SDW06.03570	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Methyl methacrylate
Certified	Yes	NJ	SDW06.03580	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Pentanone (4-methyl-2-)
Certified	Yes	NJ	SDW06.03590	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Nitropropane (2-)
Certified	Yes	NJ	SDW06.03600	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Pentachloroethane
Certified	Yes	NJ	SDW06.03610	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Propionitrile
Certified	Yes	NJ	SDW06.03615	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tert-butyl alcohol
Certified	Yes	NJ	SDW06.03620	DW	GC/MS, P & T or Direct Injection, Capillary	[EPA 524.2]	Tetrahydrofuran

Category: CLP01 -- Multi-Media, Multi-Conc. Inorganics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP01.03101	NPW	ICP/MS	[EPA ILM05.3]	Aluminum
Certified	Yes	NJ	CLP01.06101	NPW	ICP/MS	[EPA ILM05.3]	Antimony
Certified	Yes	NJ	CLP01.08101	NPW	ICP/MS	[EPA ILM05.3]	Arsenic
Certified	Yes	NJ	CLP01.11101	NPW	ICP/MS	[EPA ILM05.3]	Barium
Certified	Yes	NJ	CLP01.14101	NPW	ICP/MS	[EPA ILM05.3]	Beryllium
Certified	Yes	NJ	CLP01.19101	NPW	ICP/MS	[EPA ILM05.3]	Cadmium
Certified	Yes	NJ	CLP01.24101	NPW	ICP/MS	[EPA ILM05.3]	Chromium
Certified	Yes	NJ	CLP01.27101	NPW	ICP/MS	[EPA ILM05.3]	Cobalt
Certified	Yes	NJ	CLP01.30101	NPW	ICP/MS	[EPA ILM05.3]	Copper
Certified	Yes	NJ	CLP01.36101	NPW	ICP/MS	[EPA ILM05.3]	Lead
Certified	Yes	NJ	CLP01.41101	NPW	ICP/MS	[EPA ILM05.3]	Manganese
Certified	Yes	NJ	CLP01.47101	NPW	ICP/MS	[EPA ILM05.3]	Nickel
Certified	Yes	NJ	CLP01.51101	NPW	ICP/MS	[EPA ILM05.3]	Selenium
Certified	Yes	NJ	CLP01.54101	NPW	ICP/MS	[EPA ILM05.3]	Silver
Certified	Yes	NJ	CLP01.59101	NPW	ICP/MS	[EPA ILM05.3]	Thallium

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208 SOUTH PARK DR
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Category: CLP01 -- Multi-Media, Multi-Conc. Inorganics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP01.63101	NPW	ICP/MS	[EPA ILM05.3]	Vanadium
Certified	Yes	NJ	CLP01.66101	NPW	ICP/MS	[EPA ILM05.3]	Zinc

Category: SHW04 -- Inorganic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW04.01000	NPW	Acid Digestion/Surface and Groundwater, ICP, FLAA	[SW-846 3005A, Rev. 1, 7/92]	Metals, Total Rec and Dissolved
Certified	Yes	NJ	SHW04.01500	NPW	Acid Digestion/Aqueous Samples, ICP, FLAA	[SW-846 3010A, Rev. 1, 7/92]	Metals, Total
Dropped	No	NJ	SHW04.21000	NPW	Colorimetric	[SW-846 7196A, Rev. 1, 7/92]	Chromium (VI)
Certified	Yes	NJ	SHW04.33000	NPW	AA, Manual Cold Vapor	[SW-846 7470A, Rev. 1, 9/94]	Mercury - liquid waste

Category: SHW05 -- Organic Parameters, Prep. / Screening

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW05.01000	NPW	Separatory Funnel Extraction	[SW-846 3510C, Rev. 3, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.02000	NPW	Continuous Liquid-Liquid Extraction	[SW-846 3520C, Rev. 3, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.07000	NPW	Purge & Trap Aqueous	[SW-846 5030B, Rev. 2, 12/96]	Volatile organics

Category: SHW06 -- Organic Parameters, Chromatography

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW06.23100	NPW	GC, Headspace, FID	[OTHER J. Chrom. Sci. RSK-175]	Ethane
Certified	Yes	NJ	SHW06.23105	NPW	GC, Headspace, FID	[OTHER J. Chrom. Sci. RSK-175]	Ethene
Certified	Yes	NJ	SHW06.23110	NPW	GC, Headspace, FID	[OTHER J. Chrom. Sci. RSK-175]	Methane
Certified	Yes	NJ	SHW06.23115	NPW	GC, Headspace, FID	[OTHER J. Chrom. Sci. RSK-175]	Propane

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Category: SHW09 -- Miscellaneous Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW09.17000	NPW	Wheatstone Bridge	[SW-846 9050A, Rev. 1, 12/96]	Specific conductance
Certified	Yes	NJ	SHW09.19000	NPW	Infrared Spectrometry or FID	[SW-846 9060, Rev. 0, 9/86]	Total organic carbon (TOC)

Category: WPP02 -- Inorganic Parameters, Nutrients and Dema

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Dropped	No	NJ	WPP02.01500	NPW	Electrometric or Color Titration	[EPA 310.1]	Alkalinity as CaCO3
Certified	Yes	NJ	WPP02.02500	NPW	Distillation, Nesslerization	[EPA 350.2]	Ammonia
Dropped	No	NJ	WPP02.05000	NPW	Dissolved Oxygen Depletion	[EPA 405.1]	Biochemical oxygen demand
Certified	Yes	NJ	WPP02.06000	NPW	ICP	[EPA 200.7]	Boron
Certified	Yes	NJ	WPP02.08000	NPW	Digestion, ICP	[EPA 200.7]	Calcium
Dropped	No	NJ	WPP02.09500	NPW	Dissolved Oxygen Depletion, Nitrification Inhibition	[SM 5210 B]	Carbonaceous BOD (CBOD)
Certified	Yes	NJ	WPP02.10500	NPW	Spectrophotometric Manual/Auto	[EPA 410.4]	Chemical oxygen demand
Dropped	No	NJ	WPP02.12500	NPW	Colorimetric, Automated (Ferricyanide)	[EPA 325.1 OR .2]	Chloride
Certified	Yes	NJ	WPP02.12600	NPW	Ion Chromatography	[EPA 300.0]	Chloride
Certified	Yes	NJ	WPP02.15500	NPW	Distillation, Spectrophotometric (Auto)	[EPA 335.3] [EPA 335.4]	Cyanide
Certified	Yes	NJ	WPP02.18100	NPW	Ion Chromatography	[EPA 300.0]	Fluoride
Dropped	No	NJ	WPP02.19000	NPW	Titrimetric, EDTA	[EPA 130.2] [SM 2340 B or C]	Hardness - total as CaCO3
Certified	Yes	NJ	WPP02.20100	NPW	Ca + Mg Carbonates, ICP	[EPA 200.7]	Hardness - total as CaCO3
Certified	Yes	NJ	WPP02.21000	NPW	Digestion, Distillation, Nesslerization	[EPA 351.3]	Kjeldahl nitrogen - total
Certified	Yes	NJ	WPP02.24000	NPW	Digestion, ICP	[EPA 200.7]	Magnesium
Certified	Yes	NJ	WPP02.26100	NPW	Ion Chromatography	[EPA 300.0]	Nitrate
Certified	Yes	NJ	WPP02.27000	NPW	Cadmium Reduction, Automated	[EPA 353.2]	Nitrate - nitrite
Certified	Yes	NJ	WPP02.28000	NPW	Spectrophotometric, Manual	[EPA 354.1]	Nitrite
Certified	Yes	NJ	WPP02.30000	NPW	Combustion or Oxidation	[EPA 415.1]	Total organic carbon (TOC)
Certified	Yes	NJ	WPP02.31500	NPW	Ascorbic Acid, Manual Single Reagent	[EPA 365.2]	Orthophosphate
Certified	Yes	NJ	WPP02.35000	NPW	Auto Ascorbic Acid Reduction	[EPA 365.2 + .1]	Phosphorus (total)
Certified	Yes	NJ	WPP02.36500	NPW	Digestion, ICP	[EPA 200.7]	Potassium
Certified	Yes	NJ	WPP02.38000	NPW	Gravimetric, 103-105 Degrees C	[EPA 160.3]	Residue - total
Certified	Yes	NJ	WPP02.38500	NPW	Gravimetric, 180 Degrees C	[EPA 160.1]	Residue - filterable (TDS)

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: WPP02 -- Inorganic Parameters, Nutrients and Dema

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	WPP02.39000	NPW	Gravimetric, 103-105 Degrees C, Post Washing	[EPA 160.2]	Residue - nonfilterable (TSS)
Certified	Yes	NJ	WPP02.42500	NPW	0.45u Filtration + ICP	[EPA 200.7]	Silica - dissolved
Certified	Yes	NJ	WPP02.44000	NPW	Digestion, ICP	[EPA 200.7]	Sodium
Certified	Yes	NJ	WPP02.45500	NPW	Wheatstone Bridge	[EPA 120.1]	Specific conductance
Dropped	No	NJ	WPP02.46500	NPW	Turbidimetric	[EPA 375.4]	Sulfate
Certified	Yes	NJ	WPP02.47100	NPW	Ion Chromatography	[EPA 300.0]	Sulfate
Certified	Yes	NJ	WPP02.48000	NPW	Colorimetric (Methylene Blue)	[EPA 376.2]	Sulfides
Dropped	No	NJ	WPP02.48500	NPW	Colorimetric (Methylene Blue)	[EPA 425.1]	Surfactants
Dropped	No	NJ	WPP02.50000	NPW	Nephelometric	[EPA 180.1]	Turbidity

Category: WPP03 -- Analyze-Immediately Inorganic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	WPP03.09000	NPW	Electrometric	[EPA 150.1]	pH

Category: WPP04 -- Inorganic Parameters, Metals

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	WPP04.02000	NPW	Digestion, ICP	[EPA 200.7]	Aluminum
Certified	Yes	NJ	WPP04.04500	NPW	Digestion, ICP	[EPA 200.7]	Antimony
Dropped	No	NJ	WPP04.04600	NPW	ICP/MS	[EPA 200.8]	Antimony
Certified	Yes	NJ	WPP04.05600	NPW	Digestion, ICP	[EPA 200.7]	Arsenic
Dropped	No	NJ	WPP04.05700	NPW	ICP/MS	[EPA 200.8]	Arsenic
Certified	Yes	NJ	WPP04.08000	NPW	Digestion, ICP	[EPA 200.7]	Barium
Dropped	No	NJ	WPP04.08200	NPW	ICP/MS	[EPA 200.8]	Barium
Certified	Yes	NJ	WPP04.11000	NPW	Digestion, ICP	[EPA 200.7]	Beryllium
Dropped	No	NJ	WPP04.11100	NPW	ICP/MS	[EPA 200.8]	Beryllium
Certified	Yes	NJ	WPP04.13500	NPW	Digestion, ICP	[EPA 200.7]	Cadmium
Dropped	No	NJ	WPP04.13600	NPW	ICP/MS	[EPA 200.8]	Cadmium

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208 SOUTH PARK DR
STE 1

Category: WPP04 -- Inorganic Parameters, Metals

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	WPP04.18000	NPW	Digestion, ICP	[EPA 200.7]	Chromium
Certified	Yes	NJ	WPP04.19500	NPW	Digestion, ICP	[EPA 200.7]	Cobalt
Dropped	No	NJ	WPP04.19600	NPW	ICP/MS	[EPA 200.8]	Cobalt
Certified	Yes	NJ	WPP04.21500	NPW	Digestion, ICP	[EPA 200.7]	Copper
Dropped	No	NJ	WPP04.21600	NPW	ICP/MS	[EPA 200.8]	Copper
Certified	Yes	NJ	WPP04.26500	NPW	Digestion, ICP	[EPA 200.7]	Iron
Certified	Yes	NJ	WPP04.28000	NPW	Digestion, ICP	[EPA 200.7]	Lead
Dropped	No	NJ	WPP04.28100	NPW	ICP/MS	[EPA 200.8]	Lead
Certified	Yes	NJ	WPP04.31000	NPW	Digestion, ICP	[EPA 200.7]	Manganese
Dropped	No	NJ	WPP04.31100	NPW	ICP/MS	[EPA 200.8]	Manganese
Certified	Yes	NJ	WPP04.33000	NPW	Manual Cold Vapor	[EPA 245.1]	Mercury
Certified	Yes	NJ	WPP04.35000	NPW	Digestion, ICP	[EPA 200.7]	Molybdenum
Certified	Yes	NJ	WPP04.37500	NPW	Digestion, ICP	[EPA 200.7]	Nickel
Dropped	No	NJ	WPP04.37600	NPW	ICP/MS	[EPA 200.8]	Nickel
Certified	Yes	NJ	WPP04.45500	NPW	Digestion, ICP	[EPA 200.7]	Selenium
Dropped	No	NJ	WPP04.45600	NPW	ICP/MS	[EPA 200.8]	Selenium
Certified	Yes	NJ	WPP04.48000	NPW	Digestion, ICP	[EPA 200.7]	Silver
Dropped	No	NJ	WPP04.48200	NPW	ICP/MS	[EPA 200.8]	Silver
Certified	Yes	NJ	WPP04.50000	NPW	Digestion, ICP	[EPA 200.7]	Thallium
Dropped	No	NJ	WPP04.50100	NPW	ICP/MS	[EPA 200.8]	Thallium
Certified	Yes	NJ	WPP04.51100	NPW	Digestion, ICP	[EPA 200.7]	Tin
Certified	Yes	NJ	WPP04.52050	NPW	Digestion, ICP	[EPA 200.7]	Titanium
Certified	Yes	NJ	WPP04.54000	NPW	Digestion, ICP	[EPA 200.7]	Vanadium
Dropped	No	NJ	WPP04.54100	NPW	ICP/MS	[EPA 200.8]	Vanadium
Certified	Yes	NJ	WPP04.56500	NPW	Digestion, ICP	[EPA 200.7]	Zinc
Dropped	No	NJ	WPP04.56600	NPW	ICP/MS	[EPA 200.8]	Zinc

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Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: CLP01 -- Multi-Media, Multi-Conc. Inorganics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP01.03002	NPW, SCM	ICP	[EPA ILM05.3]	Aluminum
Certified	Yes	NJ	CLP01.06002	NPW, SCM	ICP	[EPA ILM05.3]	Antimony
Certified	Yes	NJ	CLP01.08002	NPW, SCM	ICP	[EPA ILM05.3]	Arsenic
Certified	Yes	NJ	CLP01.11002	NPW, SCM	ICP	[EPA ILM05.3]	Barium
Certified	Yes	NJ	CLP01.14002	NPW, SCM	ICP	[EPA ILM05.3]	Beryllium
Certified	Yes	NJ	CLP01.19002	NPW, SCM	ICP	[EPA ILM05.3]	Cadmium
Certified	Yes	NJ	CLP01.21002	NPW, SCM	ICP	[EPA ILM05.3]	Calcium
Certified	Yes	NJ	CLP01.24002	NPW, SCM	ICP	[EPA ILM05.3]	Chromium
Certified	Yes	NJ	CLP01.27002	NPW, SCM	ICP	[EPA ILM05.3]	Cobalt
Certified	Yes	NJ	CLP01.30002	NPW, SCM	ICP	[EPA ILM05.3]	Copper
Certified	Yes	NJ	CLP01.33002	NPW, SCM	ICP	[EPA ILM05.3]	Iron
Certified	Yes	NJ	CLP01.36002	NPW, SCM	ICP	[EPA ILM05.3]	Lead
Certified	Yes	NJ	CLP01.38002	NPW, SCM	ICP	[EPA ILM05.3]	Magnesium
Certified	Yes	NJ	CLP01.41002	NPW, SCM	ICP	[EPA ILM05.3]	Manganese
Certified	Yes	NJ	CLP01.42101	NPW, SCM	CVAA, Manual	[EPA ILM05.3]	Mercury
Certified	Yes	NJ	CLP01.47002	NPW, SCM	ICP	[EPA ILM05.3]	Nickel
Certified	Yes	NJ	CLP01.49002	NPW, SCM	ICP	[EPA ILM05.3]	Potassium
Certified	Yes	NJ	CLP01.51002	NPW, SCM	ICP	[EPA ILM05.3]	Selenium
Certified	Yes	NJ	CLP01.54002	NPW, SCM	ICP	[EPA ILM05.3]	Silver
Certified	Yes	NJ	CLP01.56002	NPW, SCM	ICP	[EPA ILM05.3]	Sodium
Certified	Yes	NJ	CLP01.59002	NPW, SCM	ICP	[EPA ILM05.3]	Thallium
Certified	Yes	NJ	CLP01.63002	NPW, SCM	ICP	[EPA ILM05.3]	Vanadium
Certified	Yes	NJ	CLP01.66002	NPW, SCM	ICP	[EPA ILM05.3]	Zinc
Certified	Yes	NJ	CLP01.69101	NPW, SCM	Midi Distillation, Spectrophotometric	[EPA ILM05.3]	Cyanide, Total in Water and Soil / Sediments

Category: CLP02 -- Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.01012	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Aldrin
Certified	Yes	NJ	CLP02.01022	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Alpha BHC

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208 SOUTH PARK DR
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Category: CLP02 -- Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.01032	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Beta BHC
Certified	Yes	NJ	CLP02.01042	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Delta BHC
Certified	Yes	NJ	CLP02.01052	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Lindane (gamma BHC)
Certified	Yes	NJ	CLP02.01062	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Chlordane (alpha)
Certified	Yes	NJ	CLP02.01072	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Chlordane (gamma)
Certified	Yes	NJ	CLP02.01082	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	DDD (4,4'-)
Certified	Yes	NJ	CLP02.01092	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	DDE (4,4'-)
Certified	Yes	NJ	CLP02.01102	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	DDT (4,4'-)
Certified	Yes	NJ	CLP02.01112	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Dieldrin
Certified	Yes	NJ	CLP02.01122	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endosulfan I
Certified	Yes	NJ	CLP02.01132	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endosulfan II
Certified	Yes	NJ	CLP02.01142	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endosulfan sulfate
Certified	Yes	NJ	CLP02.01152	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endrin
Certified	Yes	NJ	CLP02.01162	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endrin aldehyde
Certified	Yes	NJ	CLP02.01172	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Endrin ketone
Certified	Yes	NJ	CLP02.01182	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Heptachlor
Certified	Yes	NJ	CLP02.01192	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Heptachlor epoxide
Certified	Yes	NJ	CLP02.01202	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Methoxychlor
Certified	Yes	NJ	CLP02.01212	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	Toxaphene
Certified	Yes	NJ	CLP02.01232	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1016
Certified	Yes	NJ	CLP02.01242	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1221
Certified	Yes	NJ	CLP02.01252	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1232
Certified	Yes	NJ	CLP02.01262	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1242
Certified	Yes	NJ	CLP02.01272	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1248
Certified	Yes	NJ	CLP02.01282	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1254
Certified	Yes	NJ	CLP02.01292	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB 1260
Certified	Yes	NJ	CLP02.01302	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB-1262
Certified	Yes	NJ	CLP02.01312	NPW, SCM	Extraction/GC (ECD)	[EPA SOM01.0 (8/2004)]	PCB-1268
Certified	Yes	NJ	CLP02.03022	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Benzene
Certified	Yes	NJ	CLP02.03026	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Bromochloromethane
Certified	Yes	NJ	CLP02.03032	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Chlorobenzene
Certified	Yes	NJ	CLP02.03042	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorobenzene (1,2-)

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208 SOUTH PARK DR
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Category: CLP02 -- Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.03052	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorobenzene (1,3-)
Certified	Yes	NJ	CLP02.03062	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	CLP02.03066	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dioxane (1,4-)
Certified	Yes	NJ	CLP02.03072	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Ethylbenzene
Certified	Yes	NJ	CLP02.03082	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Isopropylbenzene
Certified	Yes	NJ	CLP02.03088	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichlorobenzene (1,2,3-)
Certified	Yes	NJ	CLP02.03092	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichlorobenzene (1,2,4-)
Certified	Yes	NJ	CLP02.03102	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Styrene
Certified	Yes	NJ	CLP02.03112	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Toluene
Certified	Yes	NJ	CLP02.03116	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Xylene (m- + p-)
Certified	Yes	NJ	CLP02.03118	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Xylene (o-)
Certified	Yes	NJ	CLP02.03142	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Bromodichloromethane
Certified	Yes	NJ	CLP02.03152	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Bromoform
Certified	Yes	NJ	CLP02.03162	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Bromomethane
Certified	Yes	NJ	CLP02.03172	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Carbon tetrachloride
Certified	Yes	NJ	CLP02.03182	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Chloroethane
Certified	Yes	NJ	CLP02.03192	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Chloroform
Certified	Yes	NJ	CLP02.03202	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Chloromethane
Certified	Yes	NJ	CLP02.03212	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloropropene (trans-1,3-)
Certified	Yes	NJ	CLP02.03222	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	CLP02.03232	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dibromochloromethane
Certified	Yes	NJ	CLP02.03242	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dibromo-3-chloropropane (1,2-)
Certified	Yes	NJ	CLP02.03252	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorodifluoromethane
Certified	Yes	NJ	CLP02.03262	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloroethane (1,1-)
Certified	Yes	NJ	CLP02.03272	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloroethane (1,2-)
Certified	Yes	NJ	CLP02.03282	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloroethene (1,1-)
Certified	Yes	NJ	CLP02.03292	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloroethene (trans-1,2-)
Certified	Yes	NJ	CLP02.03302	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloroethene (cis-1,2-)
Certified	Yes	NJ	CLP02.03312	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloropropane (1,2-)
Certified	Yes	NJ	CLP02.03322	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Dichloropropene (cis-1,3-)
Certified	Yes	NJ	CLP02.03332	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Methylene chloride (Dichloromethane)
Certified	Yes	NJ	CLP02.03342	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Tetrachloroethane (1,1,2,2-)

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New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: CLP02 -- Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.03352	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Tetrachloroethene
Certified	Yes	NJ	CLP02.03362	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichloroethane (1,1,1-)
Certified	Yes	NJ	CLP02.03372	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichloroethane (1,1,2-)
Certified	Yes	NJ	CLP02.03382	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichloroethene
Certified	Yes	NJ	CLP02.03392	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichlorofluoromethane
Certified	Yes	NJ	CLP02.03402	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
Certified	Yes	NJ	CLP02.03412	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Vinyl chloride
Certified	Yes	NJ	CLP02.03432	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Acetone
Certified	Yes	NJ	CLP02.03442	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Carbon disulfide
Certified	Yes	NJ	CLP02.03452	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Cyclohexane
Certified	Yes	NJ	CLP02.03462	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Butanone (2-)
Certified	Yes	NJ	CLP02.03472	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Hexanone (2-)
Certified	Yes	NJ	CLP02.03482	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Methyl acetate
Certified	Yes	NJ	CLP02.03492	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Methylcyclohexane
Certified	Yes	NJ	CLP02.03502	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Pentanone (4-methyl-2-)
Certified	Yes	NJ	CLP02.03512	NPW, SCM	GC/MS/SIM, P & T, Capillary	[EPA SOM01.0 (8/2004)]	Tert-butyl methyl ether
Certified	Yes	NJ	CLP02.04022	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Atrazine
Certified	Yes	NJ	CLP02.04032	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	N-Nitrosodiphenylamine
Certified	Yes	NJ	CLP02.04042	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	N-Nitroso-di-n-propylamine
Certified	Yes	NJ	CLP02.04052	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Carbazole
Certified	Yes	NJ	CLP02.04062	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorobenzidine (3,3'-)
Certified	Yes	NJ	CLP02.04072	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Chloroaniline (4-)
Certified	Yes	NJ	CLP02.04082	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitroaniline (2-)
Certified	Yes	NJ	CLP02.04092	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitroaniline (3-)
Certified	Yes	NJ	CLP02.04102	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitroaniline (4-)
Certified	Yes	NJ	CLP02.04122	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Chloronaphthalene (2-)
Certified	Yes	NJ	CLP02.04132	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Hexachlorobenzene
Certified	Yes	NJ	CLP02.04142	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Hexachlorobutadiene (1,3-)
Certified	Yes	NJ	CLP02.04152	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Hexachlorocyclopentadiene
Certified	Yes	NJ	CLP02.04162	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Hexachloroethane
Certified	Yes	NJ	CLP02.04182	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Bis (2-chloroethoxy) methane
Certified	Yes	NJ	CLP02.04192	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Bis (2-chloroisopropyl) ether

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Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: CLP02 -- Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.04202	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Bis (2-chloroethyl) ether
Certified	Yes	NJ	CLP02.04212	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Chlorophenyl-phenyl ether (4-)
Certified	Yes	NJ	CLP02.04222	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Bromophenyl-phenyl ether (4-)
Certified	Yes	NJ	CLP02.04232	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitroaromatics and isophorone
Certified	Yes	NJ	CLP02.04242	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dinitrotoluene (2,4-)
Certified	Yes	NJ	CLP02.04252	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dinitrotoluene (2,6-)
Certified	Yes	NJ	CLP02.04262	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Isophorone
Certified	Yes	NJ	CLP02.04272	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitrobenzene
Certified	Yes	NJ	CLP02.04292	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Butyl benzyl phthalate
Certified	Yes	NJ	CLP02.04302	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Bis (2-ethylhexyl) phthalate
Certified	Yes	NJ	CLP02.04312	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Diethyl phthalate
Certified	Yes	NJ	CLP02.04322	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dimethyl phthalate
Certified	Yes	NJ	CLP02.04332	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Di-n-butyl phthalate
Certified	Yes	NJ	CLP02.04342	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Di-n-octyl phthalate
Certified	Yes	NJ	CLP02.04362	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Acenaphthene
Certified	Yes	NJ	CLP02.04372	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Anthracene
Certified	Yes	NJ	CLP02.04382	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Acenaphthylene
Certified	Yes	NJ	CLP02.04392	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzo(a)anthracene
Certified	Yes	NJ	CLP02.04402	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzo(a)pyrene
Certified	Yes	NJ	CLP02.04412	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzo(b)fluoranthene
Certified	Yes	NJ	CLP02.04422	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzo(ghi)perylene
Certified	Yes	NJ	CLP02.04432	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzo(k)fluoranthene
Certified	Yes	NJ	CLP02.04442	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Chrysene
Certified	Yes	NJ	CLP02.04452	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dibenzo(a,h)anthracene
Certified	Yes	NJ	CLP02.04462	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Fluoranthene
Certified	Yes	NJ	CLP02.04472	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Fluorene
Certified	Yes	NJ	CLP02.04482	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Indeno(1,2,3-cd)pyrene
Certified	Yes	NJ	CLP02.04492	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Methylnaphthalene (2-)
Certified	Yes	NJ	CLP02.04502	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Naphthalene
Certified	Yes	NJ	CLP02.04512	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Phenanthrene
Certified	Yes	NJ	CLP02.04522	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Pyrene
Certified	Yes	NJ	CLP02.04542	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Methyl phenol (4-chloro-3-)

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Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: CLP02 -- Multi-Media, Multi-Conc. Organics

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	CLP02.04552	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Chlorophenol (2-)
Certified	Yes	NJ	CLP02.04562	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dichlorophenol (2,4-)
Certified	Yes	NJ	CLP02.04572	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dimethylphenol (2,4-)
Certified	Yes	NJ	CLP02.04582	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dinitrophenol (2,4-)
Certified	Yes	NJ	CLP02.04592	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dinitrophenol (2-methyl-4,6-)
Certified	Yes	NJ	CLP02.04602	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Methylphenol (2-)
Certified	Yes	NJ	CLP02.04612	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Methylphenol (4-)
Certified	Yes	NJ	CLP02.04622	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitrophenol (2-)
Certified	Yes	NJ	CLP02.04632	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Nitrophenol (4-)
Certified	Yes	NJ	CLP02.04642	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Pentachlorophenol
Certified	Yes	NJ	CLP02.04652	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Phenol
Certified	Yes	NJ	CLP02.04662	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Trichlorophenol (2,4,5-)
Certified	Yes	NJ	CLP02.04672	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Trichlorophenol (2,4,6-)
Certified	Yes	NJ	CLP02.04692	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Acetophenone
Certified	Yes	NJ	CLP02.04702	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Benzaldehyde
Certified	Yes	NJ	CLP02.04712	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Biphenyl (1,1'-)
Certified	Yes	NJ	CLP02.04722	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Caprolactam
Certified	Yes	NJ	CLP02.04732	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Dibenzofuran
Certified	Yes	NJ	CLP02.04742	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Tetrachlorobenzene (1,2,4,5-)
Certified	Yes	NJ	CLP02.04752	NPW, SCM	Extraction, GC/MS/SIM, Capillary	[EPA SOM01.0 (8/2004)]	Tetrachlorophenol (2,3,4,6-)

Category: SHW02 -- Characteristics of Hazardous Waste

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Dropped	No	NJ	SHW02.01000	NPW, SCM	Pensky Martens	[SW-846 1010, Rev. 0, 9/86]	Ignitability
Certified	Yes	NJ	SHW02.06900	NPW, SCM	TCLP, Toxicity Procedure, ZHE	[SW-846 1311, Rev. 0, 7/92]	Volatile organics
Certified	Yes	NJ	SHW02.07000	NPW, SCM	TCLP, Toxicity Procedure, Shaker	[SW-846 1311, Rev. 0, 7/92]	Metals - semi volatile organics
Certified	Yes	NJ	SHW02.08000	NPW, SCM	Synthetic PPT Leachate Procedure	[SW-846 1312, Rev. 0, 9/94]	Metals - organics

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208 SOUTH PARK DR
STE 1

Category: SHW03 -- Analyze-Immediately Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW03.01000	NPW, SCM	Aqueous, Electrometric	[SW-846 9040B, Rev. 2, 1/95]	pH

Category: SHW04 -- Inorganic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW04.05000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Aluminum
Applied	No	NJ	SHW04.05500	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Aluminum
Certified	Yes	NJ	SHW04.06500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Antimony
Certified	Yes	NJ	SHW04.07000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Antimony
Certified	Yes	NJ	SHW04.09000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Arsenic
Certified	Yes	NJ	SHW04.09500	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Arsenic
Certified	Yes	NJ	SHW04.11500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Barium
Certified	Yes	NJ	SHW04.12000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Barium
Certified	Yes	NJ	SHW04.13500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Beryllium
Certified	Yes	NJ	SHW04.14000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Beryllium
Certified	Yes	NJ	SHW04.15100	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Boron
Certified	Yes	NJ	SHW04.15500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Cadmium
Certified	Yes	NJ	SHW04.16000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Cadmium
Certified	Yes	NJ	SHW04.17500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Calcium
Certified	Yes	NJ	SHW04.18500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Chromium
Certified	Yes	NJ	SHW04.19000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Chromium
Certified	Yes	NJ	SHW04.22500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Cobalt
Certified	Yes	NJ	SHW04.23000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Cobalt
Certified	Yes	NJ	SHW04.24500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Copper
Certified	Yes	NJ	SHW04.25000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Copper
Certified	Yes	NJ	SHW04.26000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Iron
Certified	Yes	NJ	SHW04.27500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Lead
Certified	Yes	NJ	SHW04.28000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Lead
Certified	Yes	NJ	SHW04.30500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Magnesium
Certified	Yes	NJ	SHW04.31500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Manganese

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208 SOUTH PARK DR
STE 1

Category: SHW04 -- Inorganic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW04.31600	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Manganese
Certified	Yes	NJ	SHW04.34000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Molybdenum
Certified	Yes	NJ	SHW04.35500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2, 12/96]	Nickel
Certified	Yes	NJ	SHW04.36000	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 7/92]	Nickel
Certified	Yes	NJ	SHW04.38000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Potassium
Certified	Yes	NJ	SHW04.39000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Selenium
Certified	Yes	NJ	SHW04.40600	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Selenium
Certified	Yes	NJ	SHW04.41000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Silver
Certified	Yes	NJ	SHW04.41500	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Silver
Certified	Yes	NJ	SHW04.43000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Sodium
Certified	Yes	NJ	SHW04.44000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Strontium
Certified	Yes	NJ	SHW04.45000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Thallium
Certified	Yes	NJ	SHW04.45500	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Thallium
Certified	Yes	NJ	SHW04.47100	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Tin
Certified	Yes	NJ	SHW04.47500	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Vanadium
Certified	Yes	NJ	SHW04.47505	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Vanadium
Certified	Yes	NJ	SHW04.49000	NPW, SCM	ICP	[SW-846 6010B, Rev. 2 12/96]	Zinc
Certified	Yes	NJ	SHW04.49500	NPW, SCM	ICP/MS	[SW-846 6020, Rev. 0, 9/94]	Zinc

Category: SHW05 -- Organic Parameters, Prep. / Screening

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Applied	No	NJ	SHW05.07200	NPW, SCM	Vacuum Distillation	[SW-846 5032, Rev. 0, 12/96]	Volatile organics

Category: SHW06 -- Organic Parameters, Chromatography

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Dropped	No	NJ	SHW06.04010	NPW, SCM	GC P&T, FID	[SW-846 8015B, Rev. 2, 12/96]	Gasoline range organic
Certified	Yes	NJ	SHW06.04500	NPW, SCM	Extraction, GC, FID	[SW-846 8015B, Rev. 2, 12/96]	Diesel range organic

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: SHW06 -- Organic Parameters, Chromatography

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW06.12010	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Aldrin
Certified	Yes	NJ	SHW06.12020	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Alpha BHC
Certified	Yes	NJ	SHW06.12030	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Beta BHC
Certified	Yes	NJ	SHW06.12040	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Delta BHC
Certified	Yes	NJ	SHW06.12050	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Lindane (gamma BHC)
Certified	Yes	NJ	SHW06.12060	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Chlordane (technical)
Certified	Yes	NJ	SHW06.12070	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Chlordane (alpha)
Certified	Yes	NJ	SHW06.12080	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Chlordane (gamma)
Certified	Yes	NJ	SHW06.12090	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	DDD (4,4'-)
Certified	Yes	NJ	SHW06.12100	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	DDE (4,4'-)
Certified	Yes	NJ	SHW06.12110	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	DDT (4,4'-)
Certified	Yes	NJ	SHW06.12120	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Dieldrin
Certified	Yes	NJ	SHW06.12130	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endosulfan I
Certified	Yes	NJ	SHW06.12140	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endosulfan II
Certified	Yes	NJ	SHW06.12150	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endosulfan sulfate
Certified	Yes	NJ	SHW06.12160	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endrin
Certified	Yes	NJ	SHW06.12170	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endrin aldehyde
Certified	Yes	NJ	SHW06.12180	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Endrin ketone
Certified	Yes	NJ	SHW06.12190	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Heptachlor
Certified	Yes	NJ	SHW06.12200	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Heptachlor epoxide
Certified	Yes	NJ	SHW06.12210	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Methoxychlor
Certified	Yes	NJ	SHW06.12220	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8081A, Rev. 1, 12/96]	Toxaphene
Certified	Yes	NJ	SHW06.13110	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1016
Certified	Yes	NJ	SHW06.13120	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1221
Certified	Yes	NJ	SHW06.13130	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1232
Certified	Yes	NJ	SHW06.13140	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1242
Certified	Yes	NJ	SHW06.13150	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1248
Certified	Yes	NJ	SHW06.13160	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1254
Certified	Yes	NJ	SHW06.13170	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB 1260
Applied	No	NJ	SHW06.13200	NPW, SCM	GC, Extraction, ECD or HECD, Capillary	[SW-846 8082, Rev. 0, 12/96]	PCB Congeners (19)
Dropped	No	NJ	SHW06.21010	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Azinphos methyl
Dropped	No	NJ	SHW06.21015	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Chloropyrifos

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: SHW06 -- Organic Parameters, Chromatography

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Dropped	No	NJ	SHW06.21020	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Demeton (o-)
Dropped	No	NJ	SHW06.21030	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Demeton (s-)
Dropped	No	NJ	SHW06.21060	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Malathion
Dropped	No	NJ	SHW06.21070	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Parathion
Dropped	No	NJ	SHW06.21080	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8141A, Rev. 1, 9/94]	Parathion methyl
Dropped	No	NJ	SHW06.23001	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Acifluorfen
Dropped	No	NJ	SHW06.23003	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Bentazon
Dropped	No	NJ	SHW06.23005	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Chloramben
Certified	Yes	NJ	SHW06.23010	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	Dalapon
Dropped	No	NJ	SHW06.23011	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	DCPA
Certified	Yes	NJ	SHW06.23020	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	Dicamba
Certified	Yes	NJ	SHW06.23021	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Dichlorprop
Certified	Yes	NJ	SHW06.23030	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	Dinoseb
Certified	Yes	NJ	SHW06.23040	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	D (2,4-)
Certified	Yes	NJ	SHW06.23041	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	DB (2,4-)
Certified	Yes	NJ	SHW06.23050	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	T (2,4,5-)
Certified	Yes	NJ	SHW06.23060	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	TP (2,4,5-) (Silvex)
Certified	Yes	NJ	SHW06.23061	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Dichlorobenzoic acid (3,5-)
Certified	Yes	NJ	SHW06.23063	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	MCPA
Certified	Yes	NJ	SHW06.23064	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	MCPP
Certified	Yes	NJ	SHW06.23065	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Nitrophenol (4-)
Certified	Yes	NJ	SHW06.23066	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev. 1, 9/96]	Pentachlorophenol
Dropped	No	NJ	SHW06.23070	NPW, SCM	GC, Extraction, ECD, Capillary	[SW-846 8151A, Rev 1, 9/96]	Picloram
Certified	Yes	NJ	SHW06.28010	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	HMX
Certified	Yes	NJ	SHW06.28020	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	RDX
Certified	Yes	NJ	SHW06.28030	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Trinitrobenzene (1,3,5-)
Certified	Yes	NJ	SHW06.28040	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Dinitrobenzene (1,3-)
Certified	Yes	NJ	SHW06.28050	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Tetryl
Certified	Yes	NJ	SHW06.28060	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Nitrobenzene
Certified	Yes	NJ	SHW06.28070	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Trinitrotoluene (2,4,6-)
Certified	Yes	NJ	SHW06.28080	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Dinitrotoluene (4-amino-2,6-)
Certified	Yes	NJ	SHW06.28090	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Dinitrotoluene (2-amino-4,6-)

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Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: SHW06 -- Organic Parameters, Chromatography

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW06.28100	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Dinitrotoluene (2,4-)
Certified	Yes	NJ	SHW06.28110	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Dinitrotoluene (2,6-)
Certified	Yes	NJ	SHW06.28120	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Nitrotoluene (2-)
Certified	Yes	NJ	SHW06.28130	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Nitrotoluene (3-)
Certified	Yes	NJ	SHW06.28140	NPW, SCM	HPLC, UV Detector	[SW-846 8330, Rev. 0, 9/94]	Nitrotoluene (4-)

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.04010	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Benzene
Certified	Yes	NJ	SHW07.04011	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Bromobenzene
Certified	Yes	NJ	SHW07.04012	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Butyl benzene (n-)
Certified	Yes	NJ	SHW07.04013	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Sec-butylbenzene
Certified	Yes	NJ	SHW07.04014	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tert-butylbenzene
Certified	Yes	NJ	SHW07.04020	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chlorobenzene
Certified	Yes	NJ	SHW07.04022	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chlorotoluene (2-)
Certified	Yes	NJ	SHW07.04023	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chlorotoluene (4-)
Certified	Yes	NJ	SHW07.04030	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichlorobenzene (1,2-)
Certified	Yes	NJ	SHW07.04040	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichlorobenzene (1,3-)
Certified	Yes	NJ	SHW07.04050	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	SHW07.04060	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Ethylbenzene
Certified	Yes	NJ	SHW07.04065	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Isopropylbenzene
Certified	Yes	NJ	SHW07.04067	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Propylbenzene (n-)
Certified	Yes	NJ	SHW07.04070	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Toluene
Certified	Yes	NJ	SHW07.04071	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Isopropyltoluene (4-)
Certified	Yes	NJ	SHW07.04072	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichlorobenzene (1,2,3-)
Certified	Yes	NJ	SHW07.04073	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trimethylbenzene (1,2,4-)
Certified	Yes	NJ	SHW07.04074	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trimethylbenzene (1,3,5-)
Certified	Yes	NJ	SHW07.04080	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Xylenes (total)
Certified	Yes	NJ	SHW07.04081	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Xylene (m-)

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ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.04082	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Xylene (o-)
Certified	Yes	NJ	SHW07.04083	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Xylene (p-)
Certified	Yes	NJ	SHW07.04088	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Allyl chloride
Certified	Yes	NJ	SHW07.04089	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Bromochloromethane
Certified	Yes	NJ	SHW07.04090	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Bromodichloromethane
Certified	Yes	NJ	SHW07.04100	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Bromoform
Certified	Yes	NJ	SHW07.04110	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Bromomethane
Certified	Yes	NJ	SHW07.04115	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Butadiene (2-chloro-1,3-)
Certified	Yes	NJ	SHW07.04120	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Carbon tetrachloride
Certified	Yes	NJ	SHW07.04130	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chloroethane
Certified	Yes	NJ	SHW07.04140	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chloroethyl vinyl ether (2-)
Certified	Yes	NJ	SHW07.04150	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chloroform
Certified	Yes	NJ	SHW07.04160	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Chloromethane
Certified	Yes	NJ	SHW07.04170	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropene (trans-1,3-)
Certified	Yes	NJ	SHW07.04180	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dibromochloromethane
Certified	Yes	NJ	SHW07.04185	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dibromoethane (1,2-) (EDB)
Certified	Yes	NJ	SHW07.04186	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dibromomethane
Certified	Yes	NJ	SHW07.04187	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dibromo-3-chloropropane (1,2-)
Certified	Yes	NJ	SHW07.04190	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichlorodifluoromethane
Certified	Yes	NJ	SHW07.04200	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloroethane (1,1-)
Certified	Yes	NJ	SHW07.04210	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloroethane (1,2-)
Certified	Yes	NJ	SHW07.04220	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloroethene (1,1-)
Certified	Yes	NJ	SHW07.04230	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloroethene (trans-1,2-)
Certified	Yes	NJ	SHW07.04235	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloroethene (cis-1,2-)
Certified	Yes	NJ	SHW07.04240	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropane (1,2-)
Certified	Yes	NJ	SHW07.04241	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropane (1,3-)
Certified	Yes	NJ	SHW07.04242	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropane (2,2-)
Certified	Yes	NJ	SHW07.04249	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropene (1,1-)
Certified	Yes	NJ	SHW07.04250	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloropropene (cis-1,3-)
Certified	Yes	NJ	SHW07.04255	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dichloro-2-butene (trans-1,4-)
Certified	Yes	NJ	SHW07.04260	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Methylene chloride (Dichloromethane)
Certified	Yes	NJ	SHW07.04270	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tetrachloroethane (1,1,2,2-)

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New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.04280	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tetrachloroethene
Certified	Yes	NJ	SHW07.04282	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tetrahydrofuran
Certified	Yes	NJ	SHW07.04290	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichloroethane (1,1,1-)
Certified	Yes	NJ	SHW07.04300	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichloroethane (1,1,2-)
Certified	Yes	NJ	SHW07.04310	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichloroethene
Certified	Yes	NJ	SHW07.04320	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichlorofluoromethane
Certified	Yes	NJ	SHW07.04322	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichloro (1,1,2-) trifluoroethane (1,2,2-)
Certified	Yes	NJ	SHW07.04325	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichloropropane (1,2,3-)
Certified	Yes	NJ	SHW07.04327	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Vinyl acetate
Certified	Yes	NJ	SHW07.04330	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Vinyl chloride
Certified	Yes	NJ	SHW07.04340	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Acetone
Certified	Yes	NJ	SHW07.04350	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Carbon disulfide
Certified	Yes	NJ	SHW07.04360	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Butanone (2-)
Certified	Yes	NJ	SHW07.04367	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Ethyl methacrylate
Certified	Yes	NJ	SHW07.04370	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Hexanone (2-)
Certified	Yes	NJ	SHW07.04371	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Methacrylonitrile
Certified	Yes	NJ	SHW07.04373	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Methyl methacrylate
Certified	Yes	NJ	SHW07.04375	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Methyl iodide
Certified	Yes	NJ	SHW07.04376	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Iso-butyl alcohol
Applied	No	NJ	SHW07.04379	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Pentachloroethane
Certified	Yes	NJ	SHW07.04380	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Pentanone (4-methyl-2-)
Certified	Yes	NJ	SHW07.04385	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Propionitrile
Certified	Yes	NJ	SHW07.04390	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Methyl tert-butyl ether
Certified	Yes	NJ	SHW07.04395	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tert-butyl alcohol
Applied	No	NJ	SHW07.04398	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Acetonitrile
Certified	Yes	NJ	SHW07.04400	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Acrolein
Certified	Yes	NJ	SHW07.04410	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Acrylonitrile
Certified	Yes	NJ	SHW07.04500	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Hexachlorobutadiene (1,3-)
Certified	Yes	NJ	SHW07.04540	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260C, Rev. 2, 12/96]	Naphthalene
Certified	Yes	NJ	SHW07.04550	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Styrene
Certified	Yes	NJ	SHW07.04560	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Tetrachloroethane (1,1,1,2-)
Certified	Yes	NJ	SHW07.04570	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Trichlorobenzene (1,2,4-)

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208 SOUTH PARK DR
STE 1

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.04590	NPW, SCM	GC/MS, P & T or Direct Injection, Capillary	[SW-846 8260B, Rev. 2, 12/96]	Dioxane (1,4-)
Certified	Yes	NJ	SHW07.04665	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Acetophenone
Certified	Yes	NJ	SHW07.04670	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Acetylaminofluorene (2-)
Certified	Yes	NJ	SHW07.04675	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Aminobiphenyl (4-)
Certified	Yes	NJ	SHW07.04680	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Aramite
Certified	Yes	NJ	SHW07.04705	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chlorobenzilate
Certified	Yes	NJ	SHW07.04715	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Diallate (cis)
Certified	Yes	NJ	SHW07.04720	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Diallate (trans)
Certified	Yes	NJ	SHW07.04755	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorophenol (2,6-)
Certified	Yes	NJ	SHW07.04760	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethoate
Certified	Yes	NJ	SHW07.04767	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethylaminoazobenzene
Certified	Yes	NJ	SHW07.04770	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethylbenz(a)anthracene (7,12-)
Certified	Yes	NJ	SHW07.04775	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethyl benzidine (3,3-)
Certified	Yes	NJ	SHW07.04795	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Famphur
Certified	Yes	NJ	SHW07.04810	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Isodrin
Certified	Yes	NJ	SHW07.04815	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Isosafrole (cis-)
Certified	Yes	NJ	SHW07.04820	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Isosafrole (trans-)
Certified	Yes	NJ	SHW07.04830	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methanesulfonate (Ethyl-)
Certified	Yes	NJ	SHW07.04835	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methanesulfonate (Methyl-)
Certified	Yes	NJ	SHW07.04840	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methapyrilene
Certified	Yes	NJ	SHW07.04845	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methylcholanthrene (3-)
Certified	Yes	NJ	SHW07.04850	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Napthoquinone (1,4-)
Certified	Yes	NJ	SHW07.04855	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Napththylamine (1-)
Certified	Yes	NJ	SHW07.04860	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Napththylamine (2-)
Certified	Yes	NJ	SHW07.04870	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitroso-di-n-butylamine
Certified	Yes	NJ	SHW07.04875	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosomorpholine
Certified	Yes	NJ	SHW07.04880	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosopiperidine
Certified	Yes	NJ	SHW07.04885	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Parathion
Certified	Yes	NJ	SHW07.04890	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Parathion methyl
Certified	Yes	NJ	SHW07.04895	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pentachlorobenzene
Certified	Yes	NJ	SHW07.04900	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pentachloroethane
Certified	Yes	NJ	SHW07.04905	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pentachloronitrobenzene

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Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.04910	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phenacetin
Certified	Yes	NJ	SHW07.04920	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phenylethylamine (alpha, alpha-Dimethyl)
Certified	Yes	NJ	SHW07.04925	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phorate
Certified	Yes	NJ	SHW07.04930	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phosphorothioate (O,O,O-Triethyl)
Certified	Yes	NJ	SHW07.04940	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Picoline (2-)
Certified	Yes	NJ	SHW07.04945	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pronamide
Certified	Yes	NJ	SHW07.04950	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Quinoline -1-Oxide (4-Nitro)
Certified	Yes	NJ	SHW07.04955	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Safrole
Certified	Yes	NJ	SHW07.04960	NPW, SCM	GC, Extract or Dir Inj, NPD or FPD,Cap	[SW-846 8270C, Rev. 3, 12/96]	Sulfotepp
Certified	Yes	NJ	SHW07.04975	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Tetrachlorobenzene (1,2,4,5-)
Certified	Yes	NJ	SHW07.04980	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Tetrachlorophenol (2,3,4,6-)
Certified	Yes	NJ	SHW07.04985	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Toluidine (2-) (2-Methylaniline)
Certified	Yes	NJ	SHW07.04990	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Toluidine (5-Nitro-2-)
Certified	Yes	NJ	SHW07.05004	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosodiethylamine
Certified	Yes	NJ	SHW07.05005	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosodimethylamine
Certified	Yes	NJ	SHW07.05006	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitroso-di-n-propylamine
Certified	Yes	NJ	SHW07.05010	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosodiphenylamine
Certified	Yes	NJ	SHW07.05011	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosomethylethylamine
Certified	Yes	NJ	SHW07.05012	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	N-Nitrosopyrrolidine
Certified	Yes	NJ	SHW07.05020	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Diphenylamine
Certified	Yes	NJ	SHW07.05030	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Carbazole
Certified	Yes	NJ	SHW07.05038	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzidine
Certified	Yes	NJ	SHW07.05040	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorobenzidine (3,3'-)
Certified	Yes	NJ	SHW07.05045	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Diphenylhydrazine (1,2-)
Certified	Yes	NJ	SHW07.05048	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Aniline
Certified	Yes	NJ	SHW07.05050	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chloraniline (4-)
Certified	Yes	NJ	SHW07.05060	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitroaniline (2-)
Certified	Yes	NJ	SHW07.05062	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitroaniline (3-)
Certified	Yes	NJ	SHW07.05063	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitroaniline (4-)
Certified	Yes	NJ	SHW07.05070	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chloronaphthalene (2-)
Certified	Yes	NJ	SHW07.05080	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Hexachlorobenzene
Certified	Yes	NJ	SHW07.05090	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Hexachlorobutadiene (1,3-)

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Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.05100	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Hexachlorocyclopentadiene
Certified	Yes	NJ	SHW07.05110	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Hexachloroethane
Certified	Yes	NJ	SHW07.05115	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Hexachloropropene
Certified	Yes	NJ	SHW07.05120	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Trichlorobenzene (1,2,4-)
Certified	Yes	NJ	SHW07.05130	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Bis (2-chloroethoxy) methane
Certified	Yes	NJ	SHW07.05132	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Bis (2-chloroethyl) ether
Certified	Yes	NJ	SHW07.05140	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Bis (2-chloroisopropyl) ether
Certified	Yes	NJ	SHW07.05150	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chlorophenyl-phenyl ether (4-)
Certified	Yes	NJ	SHW07.05160	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Bromophenyl-phenyl ether (4-)
Certified	Yes	NJ	SHW07.05170	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dinitrotoluene (2,4-)
Certified	Yes	NJ	SHW07.05180	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dinitrotoluene (2,6-)
Certified	Yes	NJ	SHW07.05190	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Isophorone
Certified	Yes	NJ	SHW07.05200	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitrobenzene
Certified	Yes	NJ	SHW07.05210	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Butyl benzyl phthalate
Certified	Yes	NJ	SHW07.05220	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Bis (2-ethylhexyl) phthalate
Certified	Yes	NJ	SHW07.05230	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Diethyl phthalate
Certified	Yes	NJ	SHW07.05240	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethyl phthalate
Certified	Yes	NJ	SHW07.05250	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Di-n-butyl phthalate
Certified	Yes	NJ	SHW07.05260	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Di-n-octyl phthalate
Certified	Yes	NJ	SHW07.05270	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Acenaphthene
Certified	Yes	NJ	SHW07.05280	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Anthracene
Certified	Yes	NJ	SHW07.05290	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Acenaphthylene
Certified	Yes	NJ	SHW07.05300	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(a)anthracene
Certified	Yes	NJ	SHW07.05310	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(a)pyrene
Certified	Yes	NJ	SHW07.05320	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(b)fluoranthene
Certified	Yes	NJ	SHW07.05330	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(ghi)perylene
Certified	Yes	NJ	SHW07.05340	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(k)fluoranthene
Certified	Yes	NJ	SHW07.05350	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chrysene
Certified	Yes	NJ	SHW07.05360	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dibenzo(a,h)anthracene
Certified	Yes	NJ	SHW07.05370	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Fluoranthene
Certified	Yes	NJ	SHW07.05380	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Fluorene
Certified	Yes	NJ	SHW07.05390	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Indeno(1,2,3-cd)pyrene

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW07.05400	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methylnaphthalene (2-)
Certified	Yes	NJ	SHW07.05410	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Naphthalene
Certified	Yes	NJ	SHW07.05420	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phenanthrene
Certified	Yes	NJ	SHW07.05430	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pyrene
Certified	Yes	NJ	SHW07.05440	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methyl phenol (4-chloro-3-)
Certified	Yes	NJ	SHW07.05450	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Chlorophenol (2-)
Certified	Yes	NJ	SHW07.05460	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorophenol (2,4-)
Certified	Yes	NJ	SHW07.05470	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dimethylphenol (2,4-)
Certified	Yes	NJ	SHW07.05480	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dinitrophenol (2,4-)
Certified	Yes	NJ	SHW07.05490	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dinitrophenol (2-methyl-4,6-)
Certified	Yes	NJ	SHW07.05500	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methylphenol (2-)
Certified	Yes	NJ	SHW07.05510	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methylphenol (4-)
Certified	Yes	NJ	SHW07.05520	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitrophenol (2-)
Certified	Yes	NJ	SHW07.05530	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Nitrophenol (4-)
Certified	Yes	NJ	SHW07.05540	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pentachlorophenol
Certified	Yes	NJ	SHW07.05550	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Phenol
Certified	Yes	NJ	SHW07.05560	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Trichlorophenol (2,4,5-)
Certified	Yes	NJ	SHW07.05570	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Trichlorophenol (2,4,6-)
Certified	Yes	NJ	SHW07.05590	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Methylphenol (3-)
Certified	Yes	NJ	SHW07.05600	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dibenzofuran
Certified	Yes	NJ	SHW07.05691	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorobenzene (1,2-)
Certified	Yes	NJ	SHW07.05692	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorobenzene (1,3-)
Certified	Yes	NJ	SHW07.05700	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dichlorobenzene (1,4-)
Certified	Yes	NJ	SHW07.05710	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzoic acid
Certified	Yes	NJ	SHW07.05720	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzyl alcohol
Certified	Yes	NJ	SHW07.05750	NPW, SCM	GC/MS, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Pyridine
Applied	No	NJ	SHW07.07584	NPW, SCM	GC/MS/SIM, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(a)anthracene
Applied	No	NJ	SHW07.07586	NPW, SCM	GC/MS/SIM, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(a)pyrene
Applied	No	NJ	SHW07.07588	NPW, SCM	GC/MS/SIM, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(b)fluoranthene
Applied	No	NJ	SHW07.07590	NPW, SCM	GC/MS/SIM, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Benzo(k)fluoranthene
Applied	No	NJ	SHW07.07594	NPW, SCM	GC/MS/SIM, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Dibenzo(a,h)anthracene

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: SHW07 -- Organic Parameters, Chromatography/MS

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Applied	No	NJ	SHW07.07598	NPW, SCM	GC/MS/SIM, Extract or Dir Inj, Capillary	[SW-846 8270C, Rev. 3, 12/96]	Indeno(1,2,3-cd)pyrene

Category: SHW09 -- Miscellaneous Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW09.05000	NPW, SCM	Colorimetric, Automated	[SW-846 9012A, Rev. 1, 12/96]	Cyanide
Certified	Yes	NJ	SHW09.09000	NPW, SCM	Redox Titration	[SW-846 9030B, Rev. 2, 12/96]	Sulfides, acid sol. & insol.
Certified	Yes	NJ	SHW09.10100	NPW, SCM	Titration	[SW-846 9034, Rev. 0, 12/96]	Sulfides, acid sol. & insol.
Certified	Yes	NJ	SHW09.13000	NPW, SCM	Turbidimetric	[SW-846 9038, Rev. 0, 9/86]	Sulfate
Certified	Yes	NJ	SHW09.13050	NPW, SCM	Ion Chromatography	[SW-846 9056, Rev. 0, 9/94]	Sulfate
Certified	Yes	NJ	SHW09.14000	NPW, SCM	Electrometric	[SW-846 9040B, Rev. 2, 1/95]	pH - waste, >20% water
Applied	No	NJ	SHW09.18010	NPW, SCM	Ion Chromatography, Bomb Combustion, Solids	[SW-846 9056, Rev. 0, 9/94]	Inorganic anions
Certified	Yes	NJ	SHW09.21000	NPW, SCM	Colorimetric, Man, 4AAP Distillation	[SW-846 9065, Rev. 0, 9/86]	Phenols
Certified	Yes	NJ	SHW09.30150	NPW, SCM	Ion Chromatography	[SW-846 9056, Rev. 0, 12/94]	Nitrate
Certified	Yes	NJ	SHW09.31000	NPW, SCM	Colorimetric, Automated (Ferri-CN AAI)	[SW-846 9250, Rev. 0, 9/86]	Chloride
Certified	Yes	NJ	SHW09.33100	NPW, SCM	Ion Chromatography	[SW-846 9056, Rev. 0, 12/96]	Chloride
Certified	Yes	NJ	SHW09.34150	NPW, SCM	Ion Chromatography	[SW-846 9056, Rev. 0, 12/96]	Fluoride
Certified	Yes	NJ	SHW09.34160	NPW, SCM	Ion Chromatography	[USER DEFINED EPA 314.0 modified for NPW & SCM matrix]	Perchlorate
Certified	Yes	NJ	SHW09.34165	NPW, SCM	LC MS/MS	[USER DEFINED EPA 331.0 modified for NPW & SCM matrix]	Perchlorate

Category: SHW10 -- Facility-Specific Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Applied	No	NJ	SHW10.21100	NPW, SCM	GC/MS	[USER DEFINED No method specified]	Alkylated PAHs

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New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: SHW04 -- Inorganic Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW04.03000	SCM	Acid Digestion, Soil Sediment & Sludge	[SW-846 3050B, Rev. 2, 12/96]	Metals
Certified	Yes	NJ	SHW04.33500	SCM	AA, Manual Cold Vapor	[SW-846 7471A, Rev. 1, 9/94]	Mercury - solid waste

Category: SHW05 -- Organic Parameters, Prep. / Screening

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW05.03000	SCM	Soxhlet Extraction	[SW-846 3540C, Rev. 3, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.04000	SCM	Automatic Soxhlet Extraction	[SW-846 3541, Rev. 0, 9/94]	Semivolatile organics
Certified	Yes	NJ	SHW05.05000	SCM	Ultrasonic Extraction	[SW-846 3550B, Rev. 2, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.06000	SCM	Waste Dilution	[SW-846 3580A, Rev. 1, 7/92]	Organics
Certified	Yes	NJ	SHW05.07300	SCM	Closed System Purge & Trap	[SW-846 5035L, Rev. 0, 12/96]	Volatile organics - low conc.
Certified	Yes	NJ	SHW05.07310	SCM	Methanol Extract, Closed System P & T	[SW-846 5035H, Rev. 0, 12/96]	Volatile organics - high conc.
Certified	Yes	NJ	SHW05.12000	SCM	Cleanup-Florisil	[SW-846 3620B, Rev. 2, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.13000	SCM	Cleanup-Silica Gel	[SW-846 3630C, Rev. 3, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.14000	SCM	Cleanup-Gel Permeation	[SW-846 3640A, Rev. 1, 9/94]	Semivolatile organics
Applied	No	NJ	SHW05.15000	SCM	Cleanup-Acid/Base Partition	[SW-846 3650B, Rev. 2, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.16000	SCM	Cleanup-Sulfur Removal	[SW-846 3660B, Rev. 2, 12/96]	Semivolatile organics
Certified	Yes	NJ	SHW05.17000	SCM	Cleanup-Sulfuric Acid/KMnO4	[SW-846 3665A, Rev. 1, 12/96]	Semivolatile organics
Applied	No	NJ	SHW05.18000	SCM	Headspace, GC or GC/MS Screen	[SW-846 3810, Rev. 0, 9/86]	Volatile organics

Category: SHW09 -- Miscellaneous Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Certified	Yes	NJ	SHW09.16000	SCM	Mix with Water or Calcium Chloride	[SW-846 9045C, Rev. 3, 1/95]	pH - soil and waste

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

New Jersey Department of Environmental Protection

National Environmental Laboratory Accreditation Program

ANNUAL CERTIFIED PARAMETER LIST AND CURRENT STATUS

Effective as of 12/13/2006 until 06/30/2007

Laboratory Name: SEVERN TRENT LABORATORIES INC/STL BURLINGTON Laboratory Number: VT972 Activity ID: NLC060005
208 SOUTH PARK DR
STE 1

Category: SHW09 -- Miscellaneous Parameters

Status	Eligible to Report NJ Data	State	Code	Matrix	Technique Description	Approved Method	Parameter Description
Applied	No	NJ	SHW09.40000	SCM	Soils, Sodium Acetate	[SW-846 9081, Rev. 0, 9/86]	Cation-exchange capacity

Joseph F. Aiello, Chief

KEY: AE = Air and Emissions, BT = Biological Tissues, DW = Drinking Water, NPW = Non-Potable Water, SCM = Solid and Chemical Materials

ARCADIS BBL

Attachment 1-2

KAR Certifications

State of Michigan
Department of Environmental Quality
Environmental Science and Services Division

CERTIFIES:

KAR LABORATORIES, INC.

HAS BEEN EVALUATED AND IS APPROVED FOR THE FOLLOWING PARAMETERS FOR THE ANALYSIS OF DRINKING WATER:

FULL CERTIFICATION

ORGANIC CHEMISTRY

Regulated and Unregulated Volatile Organic Chemicals and Total Trihalomethanes
Ethylene Dibromide, and Dibromochloropropane
Chlordane, PCBs, and Toxaphene
2,4-D, 2,4,5-TP, Dalapon, Dicamba, Dinoseb, Pentachlorophenol, and Picloram
Aldrin, Alachlor, Benzo(a)pyrene, Butachlor, Di(2-ethylhexyl)adipate,
Di(2-ethylhexyl)phthalate, Dieldrin, Endrin, Heptachlor, Heptachlor Epoxide, Hexachlorobenzene,
Hexachlorocyclopentadiene, Lindane, Metolachlor, Methoxychlor, Metribuzin, Propachlor, and Simazine

This certification requires maintenance of an acceptable quality assurance program, use of approved methodology and equipment, and satisfactory performance on evaluation samples. This certification does not guarantee validity of data generated.

November 15, 2009
Expiration Date

December 5, 2006
Issuance Date

Sandra K Gregg (acting)
Chief, Laboratory Services

Laboratory Certification Officer

0047
Laboratory Number

EQC 2310 12/02
Authority: Act 399, PA 1976

State of Michigan
Department of Environmental Quality
Environmental Science and Services Division

CERTIFIES:

KAR LABORATORIES, INC.

HAS BEEN EVALUATED AND IS APPROVED FOR THE FOLLOWING PARAMETERS FOR THE ANALYSIS OF DRINKING WATER:

FULL CERTIFICATION

INORGANIC CHEMISTRY

Bromide, Fluoride, Nitrate, Nitrite, and Sulfate
Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Copper, Nickel, Selenium, Sodium, and Thallium
Mercury
Lead
Total Organic Carbon
Cyanide

This certification requires maintenance of an acceptable quality assurance program, use of approved methodology and equipment, and satisfactory performance on evaluation samples. This certification does not guarantee validity of data generated.

November 15, 2009
Expiration Date

Sandra K Gregg (acting)
Chief, Laboratory Services

December 5, 2006
Issuance Date

Laboratory Certification Officer

0047
Laboratory Number

EQC 2310 12/02
Authority: Act 399, PA 1976

State of Michigan
Department of Environmental Quality
Environmental Science and Services Division

CERTIFIES:

KAR LABORATORIES, INC.

HAS BEEN EVALUATED AND IS APPROVED FOR THE FOLLOWING PARAMETERS FOR THE ANALYSIS OF DRINKING WATER:

FULL CERTIFICATION

MICROBIOLOGY

Total Coliform and E.coli
Heterotrophic Plate Count

This certification requires maintenance of an acceptable quality assurance program, use of approved methodology and equipment, and satisfactory performance on evaluation samples. This certification does not guarantee validity of data generated.

November 15, 2009
Expiration Date

Sandra K. Gregg (acting)
Chief, Laboratory Services

December 5, 2006
Issuance Date

Laboratory Certification Officer

0047
Laboratory Number

EQC 2310 12/02
Authority: Act 399, PA 1976

ARCADIS BBL

Attachment 1-3

Laboratory Quality Assurance Management Plans and SOPs (included on CD only)

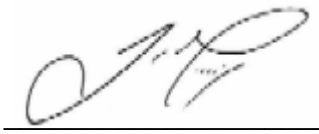
ARCADIS BBL

Attachment 1-3

STL Quality Assurance Management Plan

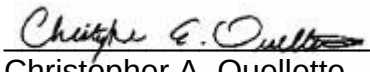
LABORATORY QUALITY MANUAL
Revision 24

Approved By:



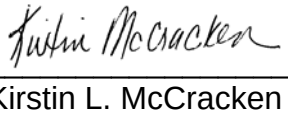
Date: March 1, 2006

Scott Morris
General Manager



Date: March 1, 2006

Christopher A. Ouellette
Laboratory Director



Date: March 1, 2006

Kirstin L. McCracken
Quality Assurance Manager

Address & Phone Number of Approved Signatories:

Severn Trent Laboratories Inc. / STL Burlington
208 South Park Drive Suite 1
Colchester, VT 05446
(802) 655-1203

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1. Introduction, Purpose and Scope

1.1. Laboratory Overview

STL Burlington located in Colchester, Vermont was founded in 1970 in response to a need for hydrographic studies in support of the nuclear power industry and water quality testing. Since its inception, the laboratory has supported environmental investigation and remediation projects all over the world with high quality, defensible data and customer-oriented service.

In 1998 STL Burlington became part of Severn Trent Laboratories, Inc. (STL). STL is the largest environmental laboratory company in the world with revenues in excess of $200 million and 29 laboratory locations in 20 states nationwide. These facilities have the combined experience of over 500 years in the environmental testing business. Through continued investment in facilities, equipment, methods and people, STL has developed an unprecedented team of resources, experience, and capabilities. With over 2000 chemists, microbiologists and environmental scientists, STL is well positioned to support a variety of clients including commercial, governmental and chemical industries.

STL's testing capabilities include chemical, physical, and biological analyses of a variety of matrices, including aqueous, solid, drinking water, waste, tissue, air and saline/estuarine samples. Specialty capabilities include dioxin and furan analysis, air, radiological testing, geotechnical testing, tissue preparation and analysis, aquatic toxicology, asbestos analysis, microscopy services, High Resolution Mass Spectrometry (HRMS), Inductively Coupled Plasma (ICP-MS), Mycology (mold and fungus) PCR microbiology, and on-site technologies including mobile laboratories. A list of STL facility locations and contact information is provided in Table 1.

STL Burlington offers a wide range of specialty services in addition to routine organic and inorganic methods, that include air toxic and sediment testing, explosives analyses for low-level reporting and extended target compound lists, PCB Congeners, Inductively Coupled Plasma (ICP-MS), and geotechnical testing. A list of the laboratory's analytical capabilities is provided in Table 2.

The laboratory provides services under various state and local municipal guidelines and operates under the rules and regulations of the following federal programs:

- Clean Air Act (CAA)
- Clean Water Act (CWA)
- Comprehensive Environmental Response Compensation and Liability Act (CERCLA)
- National Pollution Discharge Elimination System (NPDES)
- Resource Conservation and Recovery Act (RCRA)
- Safe Drinking Water Act (SDWA)
- US Army Corps Of Engineers (USACE)
- Naval Facilities Engineering Service Center (NFESC)
- National Environmental Laboratory Accreditation Program (NELAC)

A list of the certifications and approvals held by the laboratory is provided in Table 3.

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Original and current certificates are posted in the lobby of the laboratory. Original non-current certificates are archived by the QA department.

1.2. Purpose and Scope

The purpose of this Laboratory Quality Manual (LQM) is to describe the laboratory's quality system and to outline how that system enables all employees to meet STL policy, objectives, vision and mission for quality assurance. The LQM describes specific quality system activities, requirements and prescribes their frequencies. The roles and responsibilities of laboratory management and staff in support of the quality system are also defined in the LQM. The policies and procedures in the LQM describe how those activities are generally performed; a more detailed description of the activity may be found in laboratory standard operating procedures (SOPs).

The requirements set forth in this LQM apply to all laboratory activities. Use of the terms "must" and "shall", denote required activities where the terms "should" and "may" denote a guideline or recommendation but the activity is not required. Use of these terms does not prohibit the use of alternate approaches or method for implementation so long as the requirement is fulfilled.

This document was written to comply with the quality system standards adopted by the National Environmental Accreditation Conference (NELAC) and with the STL Corporate Quality Management Plan (QMP). The STL QMP describes general guidelines and minimum policies and procedures under which all STL facilities must conduct their operations. A copy of the STL Corporate QMP is available from the laboratory on request.

The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. If the policies and procedures given in the STL QMP conflict with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy and the LQM will conform to those requirements. Secondarily, the laboratory will operate in compliance with documented client requirements, when they do not conflict with regulatory requirements. The laboratory will not enter into any client agreements that conflict with regulatory requirements in the jurisdiction in which the work is performed. Where documented client agreements conflict with the policies and procedures given in this LQM but meet the regulatory requirements of the jurisdiction in which the work is performed, the client agreement shall super cede the requirements in this LQM.

The LQM is reviewed annually for accuracy and adequacy and updated as appropriate. The LQM is maintained by the QA Manager and distributed under controlled-distribution to all areas of the laboratory. Laboratory management shall ensure that these policies and procedures described in this LQM are communicated to, understood and implemented by all laboratory personnel.

2. References

See Appendix A.

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3. Terms and Definitions

See Appendix B.

4. Organization and Management

4.1. Organizational Structure

The laboratory's organizational structure is provided in Figure 1. The laboratory is under the direct supervision of a General Manager who reports to the Chief Operating Officer (COO). The relationship of the General Manager to Corporate STL is provided in Figure 2.

The laboratory's Technical Director has overall responsibility for the technical operations of the laboratory and the Quality Assurance Manager has overall responsibility for the quality system and its implementation. In the absence of the Technical Director or the QA Manager, their direct supervisor will assume the responsibilities of the absent employee or delegate the responsibility to qualified personnel.

4.2. Roles and Responsibilities

Laboratory management determines the minimum level of qualification, experience and skills necessary for all positions. A complete list of job descriptions including essential duties and responsibilities, secondary duties, working relationships, and other requirements for all positions is maintained by and available upon request from the laboratory's Human Resource Coordinator.

General Manager (GM)
The General Manager reports to the Chief Operating Office and is directly responsible for the daily operations of one or more operating facilities within STL. The GM's responsibilities include allocation of personnel and resources, long term planning, setting goals and achieving the financial, business and quality objectives of STL. The GM ensures timely compliance with corporate management directives, policies and management system reviews. The GM is an approved laboratory signatory.

Laboratory Director (LD)
The Laboratory Director reports to the General Manager and oversees the daily operations of the laboratory. The LD responsibilities include supervision of staff, setting goals and objectives for both the business and the employees, and achieving the financial, business, technical and quality objectives of the laboratory. The LD ensures timely compliance with audits and corrective actions, and is responsible for maintaining a working environment that encourages open, constructive problem solving for continuous improvement. The LD also serves as the laboratory's Technical Director and is an approved laboratory signatory.

Technical Director (TD)
The Technical Director reports to the Laboratory Director and has overall responsibility for a defined portion of the technical operations of the laboratory. The Technical Director solves day to day technical issues, provides technical training and guidance to laboratory staff, project

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managers, and clients, investigates technical issues identified by QA, and directs evaluation of new methods.

QA Manager (QAM)

The QA Manager reports to the Laboratory Director and has an indirect reporting relationship to the STL Corporate QA Director. The QAM is independent of laboratory operations and is responsible for the development and implementation of the laboratory quality system. The QAM responsibilities include ensuring that the laboratory's quality system and LQM meets the requirements set forth in the STL QMP, providing quality systems training to all new personnel, maintaining the Laboratory Quality Manual (LQM) and standard operating procedures (SOPs), and performing or overseeing systems, data, special and external audits. The QAM performs or supervises the maintenance of QA records, the maintenance of certifications and accreditations, the submission of monthly QA reports, and assists in reviewing new work as needed. The QAM has the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QAM is available to any employee at the facility to resolve data quality or ethical issues. The QAM is an approved laboratory signatory.

Customer Service Manager (CSM)

The Customer Service Manager reports to the Laboratory Director and is responsible for supervision of the project management staff. The CSM compiles and interprets the receipts forecast and tracks and maintains information for various revenue reports. The CSM is responsible for the evaluation and preparation of bids and proposals for new business opportunities and overseeing the project management bid activity for existing client base.

Project Manager (PM)

The Project Manager(s) reports to the Customer Service Manager and is responsible for direct communication with the client, coordination of laboratory services, work scheduling and dissemination of project requirements to the laboratory operation. The PM writes project narratives, performs tertiary data review, investigates and resolves technical and service related issues that arise during the course of the project.

Department Manager / Supervisor / Coordinator

The Department Manager(s) reports to the Operations Manager. The Department Manager has responsibilities for a defined portion of the laboratory that include work scheduling, development, execution and supervision of analytical procedures including SOP review and revision, secondary data review, staff training, goal setting and monitoring lab activities to achieve the quality objectives set forth in the LQM and standard operating procedures. A department supervisor or coordinator may be designated by the Department Manager to perform some of these job responsibilities. Department Supervisors or Coordinators report to the Department Manager.

Chemist / Analyst

Chemists and analysts report to the respective Department Manager or Supervisor and are responsible for analysis of samples and generation of analytical data in accordance with the

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requirements set forth in the LQM, written standard operating procedures, and project specifications.

Sample Custodian

The Sample Custodian(s) report to the respective Section Manager and is responsible for the receipt and handling of samples within the laboratory. Responsibilities include adherence to the laboratory sample acceptance policy, initiation of internal chain of custody, when needed, sample log-in and tracking, sample security and storage, and sample disposal.

Information Technology

The IT Staff are responsible for the design and maintenance of the laboratory's computer hardware and software. Responsibilities include preparation and maintenance of the Information Systems Quality Manual (ISQM), implementation and validation of new data systems, network administration, hardware and software maintenance, review, generation and implementation of electronic data deliverables (EDD) and the provision of technical support and training to all laboratory staff.

Environmental Health & Safety Coordinator

The Employee Health and Safety Coordinator is responsible for administering the EH&S program in order to provide a safe, healthy working environment for all employees. The EH&S Coordinator responsibilities include the monitoring of all work areas to detect unsafe conditions, acts, and potential hazards, enforcement of environmental, health, and safety policies and procedures and ensuring regulatory compliance with local, state, and federal laws. The EH& S Coordinator provides safety and health recommendations to laboratory management in conjunction with the facility safety committee, develops the facility Integrated Contingency Plan and coordinates the facility's Emergency Response Team.

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Figure 1:

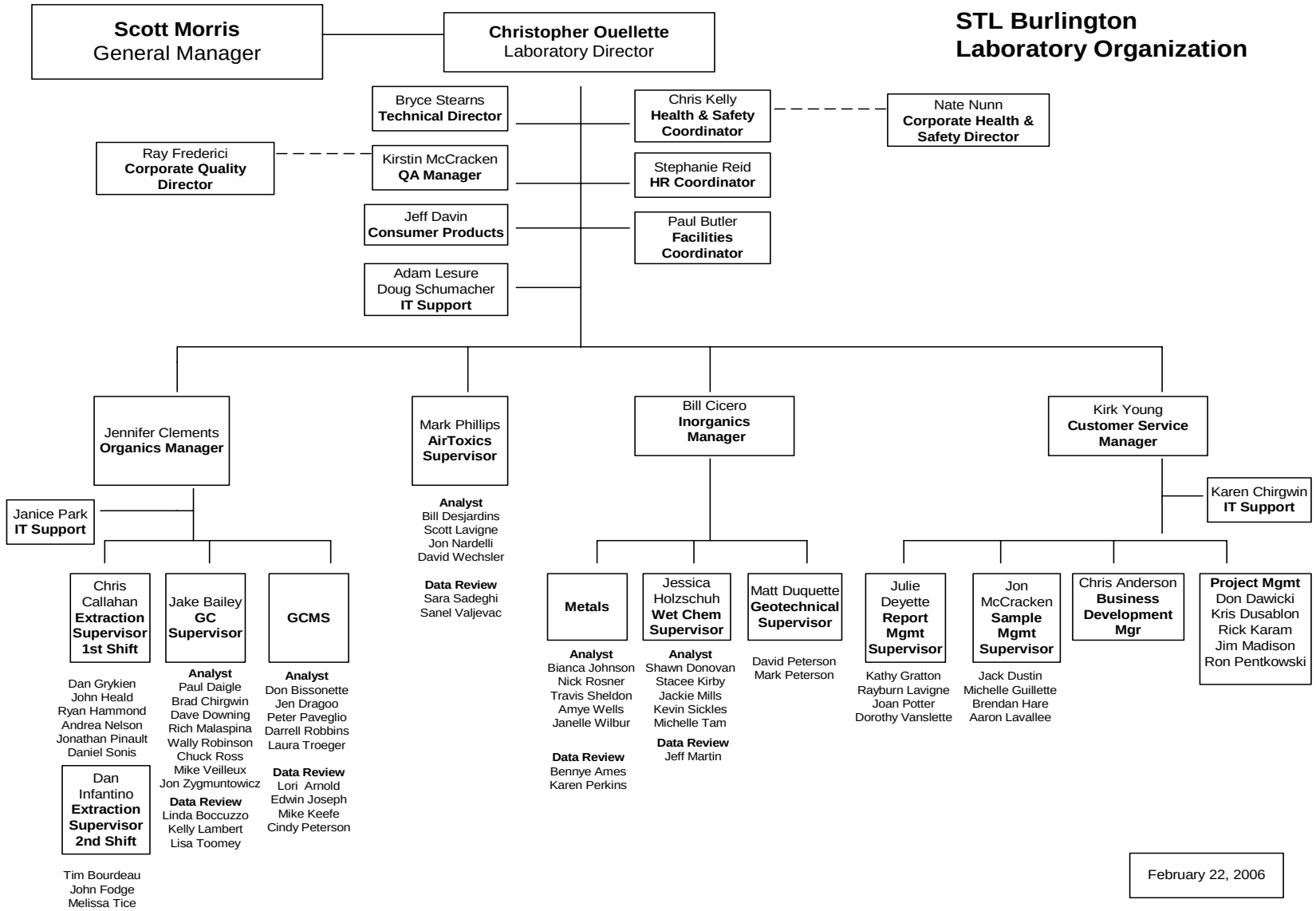

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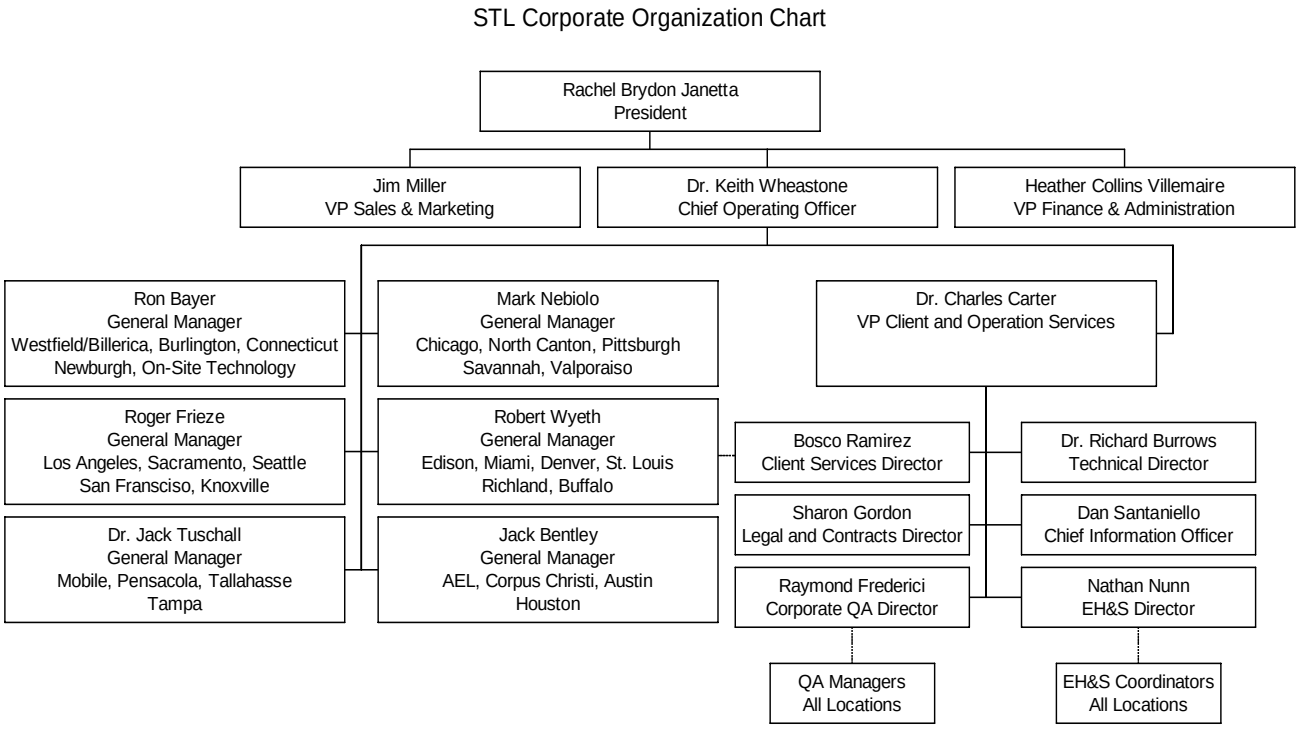
Figure 2: STL Corporate Organization Chart



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5. Quality System

5.1. Quality System Objectives

The goal of the laboratory's quality system is to ensure that business and technical operations are conducted with the highest standards of professionalism and ethics in the industry. To achieve this goal, it is necessary to provide our clients with the highest quality of service available in the industry as well as scientifically sound, documented, regulatory compliant data. The laboratory's quality system is designed to minimize systematic error, encourage constructive, documented problem solving, and to provide a framework for continuous improvement within the organization.

5.2. Quality Assurance Policy

The STL Quality Assurance Policy Statement is given in the STL Quality Management Plan (QMP) and is issued under the authority of the STL President and Chief Executive Officer, Senior Vice President and Chief Operating Officer, the Vice President of Client and Operation Services and the Director of Quality Assurance. The policy is affirmed in the laboratory as follows:

It is the Quality Assurance Policy of STL Burlington to:

- Provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- Generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- Provide STL clients with the highest level of professionalism and the best service practices in the industry.
- Build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- Maintain a working environment that fosters open communication with both clients and staff.

5.3. Commitment to Quality Assurance

Laboratory management is committed to providing the highest quality data and the best service in the environmental testing industry. To ensure that the data produced and reported by the laboratory meet the requirements of its clients and complies with the letter and spirit of municipal, state and federal regulations, STL Burlington will maintain a quality system that is clear, effective, well communicated, and supported at all levels in the company.

STL Vision & Mission Statement

STL will be the recognized leader for environmental analysis. Through the innovation and dedication of our people, together with the quality of our systems, we will deliver levels of performance that delight our clients, retain the confidence of our stakeholders and enable profitable growth of our business.

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The staff at STL Burlington is committed to providing quality data and will endeavor to use acceptable quality control and quality assurance practices for all field sampling and analytical procedures. All employees must agree to follow this Laboratory Quality Manual, corporate and laboratory policies and standard operating procedures (SOPs) and they must comply with all quality assurance/quality control (QA/QC) requirements that pertain to their job function.

5.4. Ethics and Data Integrity

The single most important aspect in assuring the highest level of data quality and service in the industry is a highly qualified, ethical and professional laboratory staff.

All employees receive training in quality assurance, ethics, data integrity, and environmental health and safety procedures and documentation of this training must be available in their employee training file and/or HR file.

This training should be performed within the time frame given in the following table:

Required Training	Time Frame*	Responsibility
Ethics Orientation	1 Day	Lab Director / QA Manager
Comprehensive Ethics Training	2 Weeks	Lab Director / QA Manager
Refresher Ethics Training	Annually	Lab Director / QA Manager
Environmental Health and Safety	1 Month	EHS Coordinator
Quality Assurance	3 Months	QA Manager

**From date of hire unless otherwise indicated.*

5.5. Ethics Policy

To ensure that all employees understand the importance the company places on maintaining high ethical standards at all times; STL has established an Ethics Policy (P-L-006) that is communicated to and understood by all laboratory personnel. Upon hire each employee watches the STL Ethics Orientation Presentation and signs the STL Ethics Agreement signifying agreed compliance with its stated purpose. Within two weeks of hire each employee should also attend a comprehensive ethics training course presented by the Laboratory Director or the QA Manager. This training includes a description of the legal and environmental repercussions that result from data misrepresentation. Additionally all employees receive annual refresher training to endorse the requirements of the ethics policy.

Data integrity is monitored by various measures that include but are not limited to; secondary and tertiary data review, internal audits including data authenticity audits, electronic data audits and special audits. STL also maintains a data integrity line administered by the STL Corporate QA Director and this direct line may be used to confidentially report suspected ethical issues or concerns. Additional procedures for confidential reporting of data integrity issues are provided in training materials distributed to each employee during ethics orientation and comprehensive ethics training. Laboratory management conducts investigations of suspected ethical misconduct under the guidance of corporate management. Violations of the ethics policy will not be tolerated. Employees who violate the policy will be subject to disciplinary action up to and

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including termination. Criminal violations may also be referred to the government for prosecution.

5.6. Personnel and Training

Laboratory management is committed to furthering the professional and technical development of employees at all levels. To achieve this goal, group training courses and workshops may be held on a variety of business, safety, laboratory procedures and technical topics; and one-on-one technical training is performed to ensure method comprehension.

Laboratory management ensures that the training for each employee is kept up to date and performed in a timely manner and that all laboratory personnel are adequately supervised.

Employee training is considered up to date if their training file(s) contain documentation that they have read, understood and agreed to follow the latest version of the LQM and the most recent version of the SOP for a given test method or laboratory process. Additionally, the training file(s) must include current documentation of Ethics, Quality Assurance, and Safety training. Each technical employee shall also have documentation of initial demonstration of capability (IDOC) and continued proficiency (DOC) for each test method performed.

Each technical employee must complete the IDOC prior to independent analysis on client samples. The IDOC study may be performed by analysis of four replicate QC samples with acceptable results for precision and accuracy. Additionally, each technical employee must demonstrate continued proficiency at least once per year by one of the following:

- Acceptable performance of a blind sample;
- Another demonstration of capability:
- Successful analysis of a blind performance sample on a similar test method using the same technology.
- Analysis of at least four consecutive laboratory control samples with acceptable levels of precision and accuracy.

If the above options cannot be performed (i.e. spiking is not an option), analysis of field samples with results that are statistically indistinguishable from those obtained by another analyst may be used to satisfy the requirement for ongoing proficiency. A certification statement signifying the analyst has met the requirements for initial and on-going proficiency shall be prepared and signed by laboratory management and maintained in the employee file.

The laboratory's training program and demonstration of capability procedures are further described in SOP LP-QA-0011 *Employee Training and Demonstration of Capability.*

5.7. Document Control

All documents relevant to the quality system are reviewed and approved by the QA Department

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prior to use in accordance with the procedures given in laboratory SOP LP-QA-0003 *Document Control.*

The following documents are issued under controlled distribution to ensure that all copies of a given document are from the latest applicable revision:

- Standard Operating Procedure
- Laboratory Quality Manual
- Quality Management Plan

Unambiguous identification of controlled documents is maintained by one or more of the following items in the document header or footer: Document Name, Document Number, Revision Number, Effective Date and Number of Pages. Documents issued under controlled distribution such as laboratory standard operating procedures (SOPs), the Laboratory Quality Manual (LQM), and the STL Corporate Quality Management Plan (QMP) will include this information in the header. Laboratory created forms used to record analytical measurements will include this information in the document footer. Additionally, laboratory logbooks are permanently bound, prenumbered, dated and distinctly labeled.

The QA Department maintains a master document list and logbook directory that identifies each document's current revision status and distribution. When a new version of a document is approved and distributed, the old version is marked obsolete and removed from the work area to prevent further use.

The STL corporate staff may issue manuals, SOPs and policies that all STL facilities are required to employ. These documents are collectively termed Official Documents and the procedures for their control, authorization and distribution is descibed in Corporate SOP S-Q-001.

5.8. Request, Tender and Contract Review

Authorized personnel perform thorough review of contract specifications for technical and quality control (QC) requirements to ensure project requirements are clearly defined and that the laboratory has the capability to meet those requirements. All contracts are reviewed to ensure that the laboratory's test methods are suitable to achieve these requirements, that the laboratory holds the appropriate certifications and approvals to perform the work, and that the appropriate environmental test method is chosen and capable of meeting the client's requirements. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements should be resolved in writing before acceptance of the contract. If the laboratory cannot provide all services but intends to subcontract such services, whether to another STL facility or to an outside firm, the intent to subcontract must be documented and discussed with the client prior to contract approval.

The laboratory shall not enter into any client agreements that conflict with regulatory requirements in the jurisdiction in which the work is performed. Where documented client agreements conflict with the policies and procedures given in this LQM but meet the regulatory requirements of the jurisdiction in which the work is performed, the client agreement shall super

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cede the requirements in this LQM.

Only appropriate personnel may approve contracts entered into by the laboratory. Contract amendments, initiated by the client and/or by the laboratory should be documented in writing for the benefit of both the client and the laboratory. All contracts, Quality Assurance Project Plans (QAPPs), Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the permanent project record.

5.9. Subcontracting

The laboratory does not routinely subcontract analytical services for which the laboratory maintains analytical capabilities as long as there is sufficient capacity to perform the work within project specifications and expected turn around time. All subcontracting is arranged with the documented consent of the client and the QC guidelines specific to the client's analytical program must be transmitted to the subcontractor and agreed upon before shipment of samples to the subcontract facility. The laboratory must obtain proof that the subcontract laboratory maintains any required certifications/accreditation and that the laboratory meets any applicable regulatory or statutory requirements for the tests to be performed. The subcontract laboratory and their work must be clearly identified in the final report.

The subcontract laboratory may receive an on-site audit by a representative of the STL QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements. The originating laboratory may also perform a paper audit of the subcontractor, which would entail reviewing the LQM, the last two PT studies, and a copy of any recent regulatory audits with the laboratory's responses. A register of verified subcontractors should be maintained along with record of evidence of compliance.

5.10. Client Complaints

An effective complaint handling process ensures that concerns are addressed promptly and provides assurance to the data user that that laboratory has confidence in its data, service obligations and products. Hearing and documenting client concerns enables the laboratory to build continuous improvement into the operation to ensure we meet our customer's needs.

All client complaints are documented, communicated to laboratory management and addressed promptly and thoroughly. The nature of the complaint is investigated and an appropriate action is determined and taken. When a client complaint indicates that an established policy or procedure was not followed, the QA Manager may conduct a special audit to assist in resolving the issue. A written confirmation, or letter to the client, outlining the issue and response taken is strongly recommended as part of the overall action taken. Monitoring the nature of client complaints and the effectiveness of resolutions is part of the laboratory's Management Systems Review process.

The laboratory procedures for handling complaints are further described in laboratory SOP LP-QA-0004 *Client Inquires, Data Validation Requests and Complaint Resolution.*

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5.11. Client Confidentiality and Proprietary Rights

Data and sample materials provided by the client or at the client's request, and the results obtained by the laboratory are be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL and client contract) subject to any disclosure required by law or legal process. Laboratory reports and the data and information provided therein, are for the exclusive use and benefit of client, and are not released to a third party without written client consent.

5.12. Service to the Client

STL Burlington assigns a Project Manager (PM) to each client to ensure that the project specific and technical requirements are effectively communicated to laboratory personnel during all project phases. The average tenure of the laboratory's PMs is over 11 years and from this base of experience, the PMs have gained extensive knowledge of regulatory programs and they understand that different projects have different data needs that may not "fit" into a standard laboratory service or product. The PMs are available to assist any client with the project planning process in order to develop a customized testing program. The laboratory also encourages clients to visit the facility and hold formal or informal meetings in order to become acquainted with laboratory staff and to provide an open forum for effective communication of project specific details.

In order for the laboratory to produce data of known quality suitable for the intended use; communication and understanding of the project data quality objectives (DQOs) and data quality indicators (DQIs) is essential.

Data Quality Objectives (DQOs) are specifications that define the project's data quality requirements based on the intended use of data. DQOs encompass both field and laboratory operations and are typically established during the development of project-specific quality assurance plans (QAPPs) and/or sampling and analysis plans (SAPs). The DQO planning process leads to the specification of sample handling procedures, sample preparation, cleanup and test methods to be used, target analyte lists, quantitation and reporting limits, use of field and laboratory QC samples, acceptance criteria, corrective action, and project needs for turn around time, data assessment, report deliverables and record retention.

Data Quality Inidcators (DQI) also known as PARCCs, precision, accuracy, representativeness, completeness and comparability are used to ensure the data produced is of known quality, appropriate for its intended use, and able to withstand scientific and legal scrutiny.

The descriptions for the DQIs given in the followings sections were derived from the STL QMP and from the Shell for Analytical Chemistry Requirements, USACE EM 200-1-3, February 2001.

Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions conform to themselves. In environmental samples, precision characterizes the natural variation of the matrix and may be used to assess matrix heterogeneity or sample handling procedures. Field precision is often determined from analysis

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of sample duplicates or matrix spikes (MS/MSD). Method precision without the effect of a matrix may be determined by analysis of data from an aggregate of laboratory control sample (LCS) results. Precision is usually expressed as relative percent difference (RPD) or relative standard deviation (RSD).

Bias refers to the systematic distortion of a measurement process that causes error in one direction and may be affected by both field and laboratory sample handling procedures. Assessment of bias is typically based on analysis of LCS, MS, MSD and surrogate standards and is commonly expressed as percent recovery (%R).

Accuracy is the degree of agreement between an observed value and an accepted reference value. Both random error (precision) and systematic error (bias) that result from sampling and analytical operations can affect accuracy. Accuracy may be determined from the analysis of LCS and MS/MSD and is typically expressed as percent recovery (R).

Representativeness refers to the degree to which data accurately and precisely characterize a population of samples, a variation in a physical or chemical property at a sampling point, or an environmental condition. Samples that are not properly collected, preserved, contaminated or analyzed beyond acceptable holding times should not be considered to provide representative data. Representativeness is primarily concerned with proper sampling and subsampling and is achieved in the laboratory by ensuring that all subsamples taken from a sample submitted for analysis is representative of the entire sample. Representativeness can be evaluated from precision of the analysis of sample duplicates and MS/MSD.

Comparability is a measure of the confidence with which one data set can be compared to another. Data sets cannot be compared with confidence unless precision and bias is known. To ensure comparability between data sets laboratory analysts shall follow approved standard operating procedures and a uniform set of units and calculations to analyze and report environmental data.

Completeness is the percentage of measurements that are judged useable compared to the total number of measurements planned or performed. The desired level of completeness is dependent on the project data quality objectives and on the size and complexity of the project.

Analytical variability (uncertainty) can be estimated when QC samples of the appropriate type and frequency are incorporated into the measurement process. The laboratory routinely analyzes QC samples to obtain data for comparison to DQO's and DQI's established for the project to ensure that the measurement system is functioning within specifications. The QC samples and their application are selected based on regulatory, method or client specific requirements and may include calibration blanks, instrument blanks, method blanks, laboratory control samples, calibration standards, internal and surrogate standards, sample duplicates and matrix spikes.

See Section 9.0 for a description of quality control (QC) sample requirements.

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5.13. Nonconformance and Corrective Action

A nonconformance includes any indication that the laboratory's product or service has not met the requirements of the relevant specification, contract or regulation. When departures from documented laboratory policies, procedures or project specifications occur, laboratory management is responsible for evaluating the significance of the nonconformance on the project data, policy or procedure, identifying the root cause of the nonconformance and implementing corrective action to prevent recurrence.

The corrective action process includes a thorough investigation to identify the possible cause of the nonconformance and whether the problem is due to an isolated error or a recurring error or breakdown in systems. Once the root cause is identified, the actions required to rectify the situation to the extent possible and to prevent recurrence of future and similar occurrences are identified along with the persons responsible for instituting the corrective action and the time frame for implementation. If the corrective action concerns a specific project related issue, the project manager should approve the corrective action before its implementation. All actions related to the corrective action process shall be documented and subsequently monitored by laboratory management to ensure that the corrective action was effective in correcting the problem and was implemented in the time frame specified.

Nonconformance and corrective action reports shall also be periodically reviewed by the QA Department to identify long-term trends or recurring problems.

The acceptance criteria and protocol for corrective actions when QC acceptance criteria are not met are provided in method SOPs. All laboratory employees are authorized to initiate and perform corrective action for QC failures as per the requirements given in the laboratory SOP. All QC failures and associated corrective actions must be documented in the project record and communicated to the client. Results associated with QC that does not meet acceptance criteria must be flagged with appropriate data qualifiers.

The laboratory procedures for nonconformance and correction action are further described in laboratory SOP LP-QA-016 *Preventive Action, Nonconformance and Corrective Action Procedures.*

5.14. Preventive Action

Laboratory operations are systematically reviewed to detect, analyze, and eliminate potential cause for nonconformance and to identify opportunities for improvement. When potential problems either technical or related to the quality system are identified, action plans are developed, implemented and monitored to reduce the occurrence of the risk identified. Some of the procedures used to identify potential risk or to drive continuous improvement include but are not limited to management review, internal audits and proficiency testing.

The laboratory procedures for preventive action are further described in laboratory SOP LP-QA-016 *Preventive Action, Nonconformance and Corrective Action Procedures.*

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5.15. Management Review

A QA report is prepared monthly by the QA Manager and submitted to the STL Corporate QA Director, General Manager, Laboratory Director, and the Operations Manager. This report includes statistical results and related information that are used to assess the effectiveness of the quality system. The content and format of this report is specified by the STL Corporate QA Director and typically includes the following information:

- Status of Internal / External Audits
- Status of Certifications / Approvals
- Results of Proficiency Testing
- Status of SOP Review and Update
- Status of Ethics Training
- Summary of Client Complaints / Negative Feedback and corrective actions
- Objectives achieved; objectives not achieved

Each month the STL Corporate QA Director prepares and submits to the STL Chief Operating Officer a monthly report containing a compilation of the facility QA reports statistics, information on progress of the corporate QA program, and a narrative outlining significant occurrences and/or concerns.

Each year the Laboratory Director and QA Manager perform an assessment of the quality system and testing and calibration activities to ensure continued suitability and effectiveness. Findings from the management review are used to implement corrective actions or create action plans for continuous improvement. The review is performed and documented in accordance with laboratory SOP LP-QA-0009 *QA Reports and Management Review* and takes into consideration the following:

- Suitability of policies and procedures
- Outcome of recent internal/external audits
- Results of proficiency testing
- Changes in volume and type of work with respect to laboratory capacity
- Positive/Negative feedback and client complaints
- Status of Staff training and resources
- Monthly QA Reports

All laboratory manages are responsible for ensuring that corrective action plans and continual improvement activities are carried out within agreed upon timeframes.

5.16. Internal Audits

Internal technical and systems audits are performed by trained and qualified employees under the direction of the QA Manager to verify that the laboratory operations comply with the requirements of this LQM and standard operating procedures. Technical audits are performed to verify compliance with method-specific requirements whereas systems audits verify compliance with the laboratory's quality system. If an internal audit finding casts doubt on the validity of test results, the laboratory must take immediate corrective action and initiate

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procedures for client notification and if necessary, data recall. Internal audits are scheduled and organized by the QA Manager in conjunction with laboratory management. To ensure that an effective non-biased audit will be conducted, laboratory employees are not permitted to audit their own activities.

Systems Audits

The QA Manager performs a systems audit are annually in accordance with STL Corporate SOP S-Q-002. Each area of the laboratory is audited to verify compliance with the laboratory's quality system using standardized checklists prepared by the STL Corporate QA Director. An audit report is prepared and submitted to the STL Corporate QA Manager, General Manager and the Laboratory Director. Laboratory management is required to respond with a corrective action plan and target date of completion for each finding noted and they are responsible for follow-through and verification to ensure that corrective actions to audit findings are effective and implemented within the agreed upon time frame.

Data Audits

Data audits are performed to assess the level of customer service, method compliance, regulatory compliance, accuracy and completeness of test results and reports, documentation, and adherence to established QC criteria, laboratory SOPs, technical policy, and project specific QC criteria. Data audits are accomplished by auditing electronic data (instrument audit), data authenticity audits, and by final project report review.

The work of all technical analysts are subject to a data authenticity audits to be performed by the QA Manager or a designee that is independent of laboratory operations. These audits should include the following: verification of raw data and evaluation of calculation tools and independent reproduction of final results including a comparison of electronic data to hardcopy records.

All organic instruments are subject to an electronic data audit to be performed by the QA Manager or designee that is independent of laboratory operations. The electronic audit may be accomplished using data mining software such as Mint Miner® followed by review of the chromatographic system. Electronic audits must include a spot check of manual integrations to ensure the integration is appropriate and documented in accordance with STL Policy S-Q-003 and laboratory SOP LP-QA-006 *Manual Integration*.

QA audits of final data reports are performed periodically to evaluate the effectiveness of the secondary data review process.

Records of the data audits are kept, and the number of audits performed is included in the monthly QA report. In performing data audits, it is essential that data be assessed in terms of differentiating between systematic and isolated errors. Upon noting anomalous data or occurrences in the data audits, the QA Department is responsible for seeking clarification from the appropriate personnel, ascertaining whether the error is systematic or an isolated error, and overseeing correction and/or revision of the project report if necessary. Errors found in client project reports are revised and the revision sent to the client. The QA Department is also

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responsible for assisting in the corrective action process where a data audit leads to identification of the need for permanent corrective action.

Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, proficiency testing results, data audits, systems audits, validation comments, or regulatory audits. Special audits are performed for a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

The laboratory procedures for internal audits are further described in laboratory SOP LP-QA-0010 *Internal Audits.*

External Audits

Clients and regulatory authorities may perform technical and systems audits of the laboratory as required by their program. During these audits, the laboratory will make every effort to provide the auditors with the personnel, documentation, and assistance required.

5.17. Proficiency Testing (PT)

The laboratory analyzes proficiency test (PT) samples as required in order to maintain accreditation or as per client or regulatory program requirements. The purpose of the PT program is to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria by analyzing unknown samples provided by an external source. As such PT samples must be handled and analyzed in the same manner as routine environmental samples. Whenever the laboratory reports an unacceptable result for a PT sample the cause for failure is investigated and necessary corrective actions are taken.

PT program requirements vary between federal, state and client programs but in general, the laboratory participates in 2 single blind inter-laboratory PT studies per year for each field of testing for which it maintains accreditation. Additionally, for each method certified under the Safe Drinking Water Act (SDWA), the laboratory analyzes at least one PT sample by each method certified per year and the laboratory participates in the annual DMR-QA Study for NPDES compliance monitoring. Single and double blind proficiency test samples may also be analyzed as per project specifications. All data related to proficiency testing is documented and archived in the same manner as routine field samples in accordance with laboratory standard operating procedures.

STL Burlington also participates in double blind performance evaluation study that is scheduled and coordinated by the STL Corporate QA Director. For this study, an external PT provider is contracted by STL to submit double blind samples to each STL facility and to assess the level of customer service and the accuracy of the test results reported by each STL facility. The external contractor provides a detailed report of the results to the Corporate QA Director and this report is subsequently used to assess all facets of STL operations.

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5.18. Records

Laboratory records include sufficient information in order to produce unequivocal, accurate documentation of laboratory activities. The identity of personnel involved in sampling, sample receipt, preparation, calibration and testing must be documented for each sample test result along with information pertaining to equipment and test methods used. Any changes to records must be signed or initialed and dated along with a reason for the change. All entries shall be made with in indelible waterproof ink and entries to records shall not be overwritten, erased or otherwise marked in such a way the original entry cannot be deciphered.

The following section describes the different record types assigned by the laboratory and provides an example of each. The laboratory procedures for management, storage and retention of each record type are further described in laboratory SOP LP-QA-014 *Laboratory Records.*

Technical and analytical records include original observations, derived data and any supporting information required to establish an audit trail and calibration record for each test report issued. At a minimum, the collective analytical record(s) must document the following items for each sample:

- Laboratory ID code
- Date/Time of Analysis
- Instrument Identification
- Analysis Method(s)
- Manual Calculations
- Manual Integrations
- Analyst Initials or signature
- Sample preparation, cleanup and analysis protocol
- Calibration and QC protocol
- Review, confirmation, interpretation, assessment and reporting conventions
- Standard and Reagent Traceability (origin, receipt, preparation and use)

Sample handling records include documentation of procedures to which a sample was subjected while in the laboratory's possession. These records pertain to sample receipt, identification, acceptance and/or rejection log in, preservation, storage and tracking and are generally archived on a project basis with a copy of the final report.

Support activity records include QA and project records such as, archived versions of the LQM and SOPs, laboratory certifications, proficiency test results and data, corrective action reports, audit reports and audit responses, copies of final reports, project correspondence, contracts and amendments and QAPP, SAPs and other related project documentation.

Administrative records include accounting documents, environmental health and safety documents, personnel files and records of employee qualifications, training and experience including demonstration of capability and a signature log for all individuals employed by the laboratory.

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Laboratory records may be electronic or hardcopy and they are stored and retained in such a way to ensure that they are readily retrievable, protected from loss by damage or deterioration and held secure and in confidence to the client for a minimum of 10 years from generation of the last record entry. Some regulatory programs may have longer record retention requirements than the laboratory standard. In these cases, the laboratory shall adhere to the most stringent retention requirement. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data must be marked as to whom to contact for authorization prior to destroying the data.

Hardcopy records are stored in an off-site warehouse protected against fire, theft, loss, deterioration, and vermin. The records are indexed in such a way that records are accessible on either a project or temporal basis. Access to archives is controlled and documented. Records stored only in electronic media are supported by hardware and software necessary for their retrieval. Any records stored or generated by computers must have hardcopy or write-protected backup copies. The procedures for computer and electronic data are further described in the following section:

5.19. Computer, Electronic Data Security and Traceability

Access to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data must be both controlled and recorded. There are various systems at the laboratory to which this applies, which include the Laboratory Information Management System (LIMS), as well as specific systems such as a chromatography data system.

System users are granted privileges that are commensurate with their experience and responsibilities. Computer access must be tracked by using unique login names and passwords for all employees that have access to the computer system. “General” or “multi-user” account access to computer systems that collect, analyze and process raw instrumental data, and those that manage and report data should not be permitted. Entries and changes are documented with the identity of the individual making the entry, and the time and date. Where a computer system is processing raw instrumental data, the instrument identification number is recorded. Many of these systems, such as the Target® Data System, have the capability of maintaining audit trails to track entries and changes to the data. This function shall be activated on any computer system that has that capability.

All commercially obtained software shall be verified prior to use and after version upgrade. Verification involves assessing whether the computer system accurately performs its intended function. Verification generally is accomplished by comparing the output of the program with the output of the raw data manually processed, or processed by the software being replaced. The records of the verification are required to contain the following information: software vendor, name of product, version, comparison of program output and manual output, raw data used to verify the program, date, and name of the individual performing the verification. Records of verification are retained as Information Technology (IT) QC records.

Additional procedures for computer systems and electronic records are further described in the laboratory Information Quality Systems Manual and STL corporate policies and procedures.

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Severn Trent Laboratories, Inc. will ensure that all records are maintained as required by the regulatory guidelines and per the QMP upon facility location change, ownership transfer, or bankruptcy. Upon STL facility location change, management of laboratory archives will be assumed by corporate STL. Upon ownership transfer and record retention requirements will be addressed in the ownership transfer agreement and the responsibility for maintaining those archives will be clearly established.

6. Facilities

STL Burlington is located in a modern, 36,000 square foot facility equipped with a computer-controlled ventilation system that reduces and eliminates background contamination. Access to the facility is secure and controlled by measures that include locked doors, electronic access cards and a staffed reception area. All visitors to the facility must sign-in and are escorted by laboratory personnel while they are in the facility.

The laboratory maintains sufficient workspace to ensure unencumbered work areas that include dedicated areas for sample receipt and storage, chemical and hazardous waste storage, data handling and storage, and sufficient separation between work areas when the analytical activities are incompatible. The laboratory is compliant with the current regulations of the Vermont Occupational Safety and Health Administration and is equipped with unique environmental controls that include monitoring of environmental conditions such as airflow, humidity, and temperature. The facility has a centralized high purity reverse osmosis water system and centralized gas distribution to support its analytical services. To minimize the production of hazardous waste, the organic preparation laboratory is equipped with a solvent recovery system.

7. Test Methods

The laboratory has implemented test methods that have been evaluated and found to be appropriate for the analysis of samples by the EPA and other state and federal regulatory agencies for a variety of environmental matrices and chemical parameters.

For each test method implemented the laboratory performs method validation and/or method verification as appropriate to verify that it can properly operate the method prior to the analysis of client samples. The level of verification and validation should be as extensive as required to ensure the method will satisfy the requirements for the intended use of data.

At a minimum, prior to the establishment of any method, a satisfactory demonstration of method capability is required. The laboratory should also establish the selectivity and sensitivity of the method, the linearity, range, level of detection, quantitation limit, reporting limit, and the accuracy and precision limits for each target compound. Additionally, the method should be formally documented in a standard operating procedure (SOP). If the method is a minor modification of a standard laboratory method that is already documented in an SOP, a SOP Appendix describing the specific differences in the new method may be used in place of a separate SOP.

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7.1. Standard Operating Procedures (SOPs)

The laboratory maintains standard operating procedures for each test method and for all laboratory activities that are implemented consistently in all areas of the laboratory such as, document control, data review, standard/reagent traceability, and nonconformance and corrective action procedures. Each SOP must include sufficient instructions and step-wise procedures to a level of detail that would allow a technically qualified individual to perform the activity without supposition.

Method SOPs are used to describe a specific test method. When the validated test method is based on a published reference method, the SOP shall be written to comply with the published reference method. Any modifications to the published method shall be described in the SOP but modifications that would result in non-compliance with a regulation are not permitted. Temporary deviations from a method SOP are permitted only when the deviation is documented, technically justified, authorized and accepted by the client and/or end user of the data.

The following information must be included in each Method SOP although it is not necessary that each of the bulleted items be included as a separate section.

- Identification of Test Method
- Applicable Matrix or Matrices
- Detection Limit
- Scope and Application
- Summary of the Test Method
- Definitions
- Interferences
- Safety
- Equipment and Supplies
- Reagents and Standards
- Sample Collection, Preservation, Shipment and Storage
- Quality Control
- Calibration and Standardization
- Procedure
- Calculations
- Method Performance
- Pollution Prevention
- Data Assessment and Acceptance Criteria for Quality Control Measures
- Corrective Actions for Out-of-Control Data
- Contingencies for Handling Out-of-Control or Unacceptable Data
- Waste Management
- Revision History
- References
- Tables, Diagrams, and Flowcharts
- Appendix (when required)

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Process SOPs describe laboratory activities not related to a specific test method and these SOPs may include some or all of the following information:

- Scope
- Summary
- Definitions
- Responsibilities
- Safety
- Procedure
- Revision History
- References
- Tables, Diagrams, and Flowcharts
- Appendix (when required)

All SOPs are document controlled according to the procedures given in laboratory SOP LP-QA-0003 *Document Control*. The SOPs are maintained by and made accessible to all laboratory personnel by the QA Department who is also responsible for archiving retired SOPs in accordance with the laboratory's system for record keeping. SOPs are reviewed annually for accuracy and adequacy and updated as appropriate.

8. Equipment

The laboratory maintains state of the art instrumentation to ensure that project data quality objectives and turn around times are met. All equipment is subject to rigorous checks upon its receipt, upgrade, or modification to establish that the equipment meets with the selectivity, sensitivity, accuracy, and precision required by the test method or intended use of the data. Instrument manuals are kept up to date and readily accessible to laboratory personnel and maintenance contracts may be held on specific pieces of equipment where outside service is efficient, cost-effective, and necessary for effective operation of the laboratory.

In order to gain maximum performance and minimize downtime, regular inspection, maintenance, cleaning, and servicing of all laboratory equipment is performed according to the manufacturers' recommendations.

A maintenance logbook shall be kept on all major pieces of equipment to document routine and non-routine maintenance. The maintenance record should include details of the maintenance performed along with date and initials of the analyst that performed the maintenance activity. Equipment that is not operating within specifications shall be removed from service until it has been repaired. Instruments taken out of service should be clearly identified or removed from the work area until it has been repaired. After repair, the return to analytical control must be documented in the maintenance logbook.

Maintenance contracts may be held on specific pieces of equipment where outside service is efficient cost effective and necessary for effective operation of the laboratory.

A list of major pieces of equipment held by the laboratory is provided in Table 4.

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8.1. Support Equipment

Equipment that are necessary to support laboratory operations but that are not the actual test instrument are maintained in proper working order and checked against an NIST traceable reference standard of measurement, when available over the range of use per the minimum frequency requirements given in the following table:

Analytical Support Equipment	**Frequency**
Refrigerator / Freezer Temperature Monitoring	Daily
Thermometer Calibration Check	Liquid in Glass – Annually Electronic (Battery) – Quarterly Thermistor/Thermocouple-Annually
Balance Calibration Check	Daily or before use with two Class S weights that bracket the range of use and annually by an outside service technician.
Class S Weight Sets	3 Years by outside service technician
Mechanic Volumetric Pipettes	Daily or prior to use
Drying Ovens	Before use

The performance criteria used to determine acceptability of use will vary based on the needs of the application for which the equipment is being used. In general, the laboratory has established tolerance ranges for analytical support equipment in accordance with the criteria established by the American Society for Testing and Materials (ASTM). Records of calibration checks and verification of support equipment are retained along with records of repair or maintenance and "How To" procedures for calibration or verification of specific types of support equipment are further described in various laboratory SOPs. Data records sufficient to reconstruct calibration of support equipment are retained in accordance with laboratory SOP LP-QA-014 *Laboratory Records.*

8.2. Instrument Calibration

Initial instrument calibration and continuing instrument calibration is performed to ensure that the analytical system is functioning properly and that the data is of known quality (i.e. precision, accuracy and sensitivity). Detailed procedures for instrument calibration are provided in method SOPs along with acceptance criteria and corrective actions. Guidance on the use of calibration data is also provided in STL Corporate SOP P-T-001 *Selection of Calibration Points*.

A brief description of calibration requirements is presented in the following sections. These requirements are based on various analytical methods and industry standards and they may be changed to reflect project-specific DQO's or the requirements given in a specific test method or regulatory program.

<u>Organic Analyses</u>
The initial calibration curve is established as specified in the reference method with a minimum of five standards at contiguous concentration ranges that reflect the quantitation range of routine samples. The concentration of the lowest calibration standard is above the method detection limit but at or below the laboratory's reporting limit. The accuracy of the standard solutions

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used for initial instrument calibration is verified with a second source standard obtained from a different manufacturer when available.

Any sample whose result is outside the upper limit of the calibration range is diluted and reanalyzed to bring the result within the calibration range, preferably in the upper half. Sample results are quantitated from the initial calibration and are not quantitated from any continuing calibration verification unless otherwise required by regulation, method or program.

When instrument calibration is not performed on the day of analysis, the validity of the initial calibration is verified prior to sample analysis by acceptable continuing calibration verification. The concentration of the CCV is varied within the calibration range. When external standard approach is used, a CCV is analyzed at the beginning and end of the analytical sequence. Additional CCVs are analyzed at the frequency required by the test method.

In general, the acceptance criteria for the CCV are based on percent difference/drift from the RSD of the initial calibration. When acceptance criteria are not met and corrective actions fail to produce a second immediate CCV within acceptance criteria; a new instrument calibration should be performed or performance demonstrated with 2 additional consecutive, successful CCVs. Samples affected by unacceptable calibration verification are reanalyzed after a new calibration curve has been established. However, sample results associated with an unacceptable CCV may be reported as qualified data when the acceptance criteria for the CCV fails high and there are no detects for those target compounds in the associated samples or when the acceptance criteria for the CCV fail low and the associated sample results exceed a maximum regulatory limit or decision level.

The following sections highlight calibration procedures for each type of instrumentation used:

GC/MS

Hardware tuning is performed on each GC/MS prior to calibration as specified in the applicable test method. Ion abundance acceptance criteria for VOC tuning with BFB or SVOC with DFTPP are provided in each test method SOP. Mass calibration is performed as an integral part of tuning. The tune check and calibration check must be performed according to the frequency requirement given in the published reference method.

Initial calibration (ICAL) is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector.

After the initial calibration standards are injected, a calibration curve is constructed using internal standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD of the calibration curve is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

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A midpoint calibration verification standard (CCV) is analyzed at the required interval as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples.

After the CCC and SPCC are evaluated, all target compounds are evaluated for linearity. If the %RSD is less than or equal to specified criteria, the average response factor can be used for quantitation. If the %RSD exceeds criteria a regression curve (linear, quadratic, etc.) may be used for quantitation if the correlation coefficient or coefficient of determination is greater than 0.99.

GC

Initial calibration (ICAL) is performed upon instrument startup and whenever the continuing calibration verification standard fails the acceptance criteria. A calibration curve is prepared for all target compounds with the lowest standard concentration at or below the reporting limit and the remaining standards defining the working range of the detector. After the initial calibration standards are injected, a calibration curve is constructed using external standard methodology. The analyst inspects the curves before proceeding with sample analysis. The correlation coefficient or coefficient of determination of the calibration curve must be greater than or equal to 0.99. An alternative to quantitation from a calibration curve is quantitation from an average response factor. If the %RSD is less than or equal to the acceptance criteria, the average response factor can be used for quantitation.

A midpoint calibration verification standard (CCV) is analyzed periodically as a check on the validity of the initial calibration. If the percent difference or percent drift is within the acceptance criteria, the curve is acceptable for quantitation of samples. Samples analyzed by external standard calibration require bracketing by CCV. If the CCV standard analyzed after the samples fails to meet the acceptance criteria and the response of the mid point standard is above the criteria (that is the response of the analytical system has increased), samples which have no target compounds detected above the RL may be reported as <RL, since the compounds would have been detected if present. (SW-846 Method 8000B).

Inorganic Analyses

For metals analyses, initial calibration is performed with each analytical batch and when a CCV fails or significant instrument maintenance is performed. Any sample whose result is above the high calibration standard is diluted and reanalyzed to bring the result within the calibration range. The analytical system for wet chemistry methods is calibrated per the requirements given in the reference method. For both metals and wet chemistry methods, Initial and continuing calibration blanks (ICB/CCB) are performed to verify the system is free of contamination. Additionally, a continuing calibration verification should be analyzed every 10 samples (or at the frequency given in the reference method, if more frequent), to determine if the system is working properly.

Metals

Initial calibration is established with a high-level calibration standard and a blank. The high-level standard sets the linear range of the calibration and minimally is set below the instrument's linear dynamic range. A low level standard is typically analyzed with each batch to verify the performance at the reporting limit. Alternatively, the instrument may be calibrated with three

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standards and a blank with the concentration of the low standard equal to or less than the reporting limit. Sample results that exceed the concentration of the high standard are diluted and reanalyzed.

Wet Chemistry
Calibration and standardization procedures vary depending on the instrument system and analytical methodology. In general, instrument systems are calibrated prior to sample analysis with the calibration range established by a series of standard solutions. The calibration is verified on a regular basis to ensure that the analytical system is still operating within specifications.

9. Purchasing of Services and Supplies

The evaluation and selection of suppliers and vendors is done, in part, on the basis of the quality of their products and service and, their ability to meet the demand for products on a continuous and short-term basis, past history and competitive pricing. To ensure that quality critical consumables and components conform to specified requirements, the specifications for such are provided in laboratory SOPs and laboratory management approves all purchase orders.

Chemical reagents, solvents, glassware and general supplies are ordered as needed to maintain a sufficient quantity on hand. The laboratory also maintains a consignment program with a reputable vendor of scientific equipment to ensure frequently used products are readily available.

10. Measurement Traceability and Calibration

Instruments and testing equipment that have an effect on the accuracy or validity of results are calibrated or verified on a routine basis against reference standards traceable to national reference standards, where applicable.

10.1. Reference Standards

Reference standards of measurement held by the laboratory such as Class S weight sets and NIST traceable thermometers are used for calibration purposes only and the calibration of these standards of measurement is periodically checked against a national standard of measurement per the frequency requirements given in standards established by the American Society for Testing and Materials (ASTM) or per the frequency requirements of a specific regulatory program, whichever is more stringent.

Standard reference materials are purchased from commercial sources in stock solution, when available. When a stock standard solution is not available, standard solutions may be prepared in the laboratory from neat materials. All purchased standard solutions must be received with a Certificate of Analysis. If the vendor cannot supply a Certificate of Analysis, the purity of the standard must be documented prior to use. All reagents used shall be analytical reagent grade unless otherwise required by the test method.

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The receipt, preparation, and disposition of all standard reference and reagent materials are documented in logbooks designated for this purpose. Purchased standard reference materials and reagents are uniquely identified with a Tag ID and prepared standards and reagent solutions are assigned internal lot numbers that are traceable to a preparation record.

The following labeling requirements apply for all Standards, Reagents and Reference Materials:

- Original containers must be labeled with an expiration date. Once the container is opened, the date opened must be recorded along with the initials of the person that opened the container and the newly assigned expiration date (when applicable).

- All containers of prepared reagents and standards must be labeled with the name and concentration of the material, the date of preparation, the initials of the analyst that prepared the material and the material's assigned expiration date.

Additional procedures for the receipt and preparation of reagents and standard reference materials are further described in laboratory SOPs LP-QA-0001 *Receipt and Tracking of Bulk Chemical and Standard Materials* and LP-QA-0002 *Standard Preparation.*

11. Sample Handling Procedures

11.1. Sample Containers

The laboratory routinely provides certified pre-cleaned sample containers and related supplies for a fee to any client per their request. Sample containers are packed in such a way to prevent breakage, placed in a pre-cleaned cooler and boxed for shipment along with any requested supplies such as packing materials, unfrozen gel ice packs* (*freeze at time of sampling), sampling instructions, blank chain-of-custody forms, and custody seals.

A sample container guide that provides container specifications by test method along with preservation and holding time requirements is provided in Appendix C.

11.2. Sample Collection and Transport to Laboratory

To ensure valid data it is imperative that the sample collectors are trained, familiar with, and have demonstrated proper sampling technique for the collection of samples. The laboratory strongly recommends that our clients establish a written sampling protocol that includes specific sampling instructions and documentation requirements for each type of sample collected for each project.

The following sections present general guidelines that should be used by those responsible for sample collection.

Sample collectors should be trained in sampling procedures and adhere to sampling instructions and/or standard operating procedures for each type of sample collected.

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All records must be made using indelible ink and identify the person responsible for each activity including transport of the samples to the laboratory.

At the time of collection the following information should be recorded for each sample:

- Sample Identification Number
- Public Water System ID (where applicable)
- Date and Time of Sample Collection
- Sample Type (i.e., compliance, confirmation, etc.)
- Analysis Method Required
- Sample Container Type, Size, and Preservative Type and Concentration
- Sample Volume or Weight Collected
- Name of Sampler and Contact Information
- Sampling Location (where applicable)
- Comments

After sample collection, the samples should be preserved, labeled and packed for shipment to the laboratory. A fixed label that includes the following information must be placed on each sample container: sample identification number, source of sample, preservative, collector's initials, and test method required.

The closed sample containers should be placed in the shipping cooler along with a chain-of-custody record and any other pertinent documentation. The sample containers must be packed to prevent breakage and to comply with any applicable Department of Transportation (DOT) regulations. The shipping cooler should be sealed in such a manner that any evidence of tampering would be readily evident upon receipt in the laboratory. The transfer of possession of samples from the field custodian to the shipper must be documented on the chain-of-custody record.

11.3. Chain of Custody

All samples received into the laboratory must be accompanied by a chain of custody form that documents the number and type of sample container used for each sample, the mode of collection, the collector's name, date and time of collection, site location, sample identification/description, type of preservation, requested analyses, and the transfer of possession of samples from the time of collection to receipt in the laboratory. Common carriers used to ship sample containers may identify themselves by signing the "Relinquished By" space on the chain-of-custody form. After sample collection the field sampling team should seal sample and/or shipping containers with a custody seal or custody tape to ensure that tampering will be immediately evident.

The chain-of-custody record should provide accurate, verifiable, and traceable records of sample possession and handling from sample container shipment through laboratory receipt. A sample is considered in custody if it is:

- In actual possession of the sampler or transferee
- In view after being in physical possession of the sampler or transferee

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- In a secured area, restricted to authorized personnel.
- Sealed so that sample integrity will be maintained while in possession of the sampler or transferee

All samples received into the laboratory and subsequently subcontracted to another laboratory must be shipped under chain-of-custody initiated by the originating laboratory. A copy of the original chain-of-custody form that was received with the samples must accompany the sample shipment.

11.4. Sample Receipt, Sample Acceptance Policy, Sample Handling

Sample Receipt

The laboratory's sample custodian(s) perform inspection of shipping coolers received to ensure that the contents have not been altered or tampered with during transit. If tampering of the shipping container is apparent, the sample custodian contacts the assigned laboratory project manager who consults with the client for further instructions.

Shipping coolers that arrive intact are immediately opened and the contents inspected in accordance with the procedures given in laboratory SOP LM-SM-001 *Sample Management.* The temperature of each shipping cooler is checked and considered acceptable if the arrival temperature is within the required temperature of the method specified range. For samples that require thermal preservation at a specified temperature of 4°C, the arrival temperature from just above freezing (0°C) to 6°C is generally considered acceptable, except samples for submitted for analysis by EPA Method 524.2 or EPA Method 504.1 in which case the temperature must be less than or equal to 4°C. If samples are hand delivered to the laboratory following collection, the receipt of samples is considered acceptable if there is evidence that the chilling process has begun. The results of these checks are recorded document-controlled checklists or in laboratory notebook(s) designated for this purpose. Each sample container is also verified against the information provided on the chain-of-custody form. If discrepancies or problems that would indicate the integrity of the sample(s) has been compromised are found, the sample custodian contacts the assigned laboratory project manager who consults with the client for further instructions.

Sample Acceptance Policy

To ensure the validity of laboratory data all sample shipments received in the laboratory must meet the following conditions:

- All samples must be received with a complete Chain of Custody form.

- The paperwork received with samples must include proper, full and complete documentation that includes, the sample identification and description, the location, date and time of collection, the collector's name, preservation type and any special remarks regarding the sample(s).

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- Sample containers must have a durable water resistant label with all entries made with indelible ink.

- Samples should be received in appropriate containers with adequate sample volume sufficient to perform the test requested.

- Samples should be received within the specified holding time.

The sample custodian flags samples that do not meet the above conditions and communicates the nature and substance of the nonconformance to the assigned laboratory project manager who consults with the client for further instructions. The nonconformance is documented in the analytical record and any correspondence related to decisions to proceed with analysis or reject samples is documented and retained in the project record. A discussion of samples that did not meet sample acceptance criteria is also included in the case narrative that accompanies the final data report.

Sample Log In, Preservation Checks, and Storage

After the samples have been checked, verified and accepted into the laboratory, a unique laboratory ID code is assigned to each sample and affixed to the sample container with a durable, water-resistant label and the samples are logged into the Laboratory Information Management Systems (LIMS) database. The laboratory's custom designed LIMS is used to track samples and analytical data throughout the facility and the laboratory ID code is the primary mechanism used to link the sample to its field code and sample receipt documentation.

After log in samples are placed in appropriate storage areas. Samples that require thermal preservation are stored under refrigeration at the specified temperature of $4 \pm 2^{o}C$ or when freezing is required, the samples are maintained at a temperature of $-15 \pm 5^{o}C$. The temperature of each refrigeration unit is continuously monitored to ensure the samples remain within the acceptable temperature range.

The chemical preservation of samples on checked on receipt or prior to or during sample preparation.

Sample Storage & Subsampling

All samples are stored according to the requirements outlined in the test method, away from standards, reagents and other potentially contaminating sources and stored in such a manner to prevent cross-contamination. Samples designated for volatile organic analysis are segregated from other samples and samples suspected of containing high levels of should be further isolated. Storage blanks are maintained in each refrigerator/freezer used to store volatile organic samples and analyzed weekly to verify that cross-contamination has not occurred.

Access to secure sample storage areas is limited to authorized laboratory personnel. All external doors are visually monitored by laboratory staff or kept locked. Visitors are required to sign in and wear a visitor's badge during their visit and are accompanied at all times by a staff

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member when in the laboratory. Lockable refrigerators and storage cabinets are also available for samples that require this level of security.

For most projects a secure facility is sufficient to maintain the integrity of samples. However, for projects that require legal chain of custody, the laboratory will upon request by the client, document the internal transfer of samples, extracts and digestates within the laboratory. The procedural requirements for internal chain of custody must be arranged and communicated to the laboratory at the time of project initiation.

The laboratory procedures for sample handling are further described in laboratory SOP LP-SM-001 *Sample Management.* The procedures and appropriate techniques used to obtain representative sub-samples from field samples received in the laboratory for analysis are described in each method SOP.

Sample Disposal

Unless otherwise specified by the client, the laboratory retains samples, digestates and extracts for 30 days after submission of the final data report. Some samples are required to be held for longer periods based on regulatory or client requirements and in those cases, the laboratory must adhere to the longer sample retention requirement as agreed upon with the client. Unused portions of samples that are found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work. Samples may also be returned to the client per their written request.

All samples shall be disposed of in accordance with federal, state and local regulations. The laboratory procedures for sample disposal are further described laboratory SOP LP-LB-0010 *Hazardous Waste Disposal*.

12. Quality Control

12.1. Quality Control Samples

Quality control samples (QC) are used to monitor the analytical process from sample collection through analysis. Laboratory QC such as method blanks and laboratory control samples are used to determine if the laboratory operation is within specifications (acceptance limits) where matrix specific QC such as the analysis of matrix spikes, matrix spike duplicates, sample duplicates and surrogate standards (when applicable) are used to assess the effect of the matrix on the data being generated. Laboratory QC may also provide useful information to assess matrix effects. Field QC samples such as field blanks and trip blanks are used to measure the affect field conditions have on analytical results. Table 5 presents a summary of the laboratory and field QC samples commonly used along with their recommended frequency for analysis.

Specific QC requirements are included in each test method SOP along with the associated acceptance criteria and corrective actions for when criteria are not met. The criteria used to assess QC samples may be based on default values given within the test method laboratory determined ranges (in-house control limits) or project data quality objectives. Where a method

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or regulation does not specify acceptance and/or rejection criteria, the laboratory will assess the demonstrated sensitivity, accuracy and precision of the available test methods in conjunction with the intended use of the data in order to determine appropriate acceptance criteria.

The routine QA objectives for accuracy and precision and the routine method detection limits (MDLs) for relatively clean environmental samples for each test method/analyte/matrix performed are available from the laboratory on request. When the QC requirements for a specific project, state or federal program are more stringent than those routinely used the laboratory, the laboratory will adhere to the more stringent requirements when those requirements are known.

Control charts are used to track the results of quality control measures on an on-going basis. Detailed procedures for the generation and monitoring of control charts are described in laboratory SOP LP-QA-0013 *Control Limits*. In general, control charts are established using the percent recovery data from LCS recovery data and control limits for surrogates are established from the recovery of surrogates in field samples. The average recovery and warning (2σ) and control limits (3σ) are based on at least 20 individual percent recovery values generated within a calendar year. The control chart results are evaluated to verify that method performance is within acceptable limits specified in analytical procedures and/or regulatory guidelines. From this evaluation, the control limits used to assess accuracy and precision are updated as necessary.

12.2. Data Reduction and Data Review

The procedures used for data reduction are described in each method SOP. Automated procedures for data reduction shall be tested and verified to ensure the software's ability to accurately acquire, process, manipulate, transfer, record and report data.

All data generated by the laboratory must be reviewed prior to the release of the data report in order to assure the validity of the data. The routine data review process shall encompass a minimum of three levels of review to ensure the correctness and completeness of data. QA review may be performed on a percentage of data reported but QA review is not part of the normal review process.

Primary Review (Analyst Review)

Each analyst shall be responsible for reviewing the quality of his/her work based on the guidelines given in this LQM and laboratory standard operating procedures. Primary review must be documented with the signature/initials of the reviewer and the date the review was performed.

At a minimum, the review must ensure the following:

- Sample preparation and analysis information is correct and complete.
- Analytical results are correct and complete
- Manual integrations are appropriate and documented.
- QC samples are within established control limits or otherwise qualified.
- Dilution factors are correctly recorded and applied.
- Data transfers are verified and correct.

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- Non-conformances have been properly documented and communicated.
- The appropriate SOPs and client requirements were followed.
- Internal COC documentation is correct and complete, when required.
- Documentation is complete.

Secondary Review (Peer Review)

After primary review, all data shall be reviewed by a manager, supervisor, coordinator, or data review analyst, who has documented training for the areas for which he/she provides review. This review shall be a complete, independent review of the entire analytical data report and shall be performed so that all calibration data, QC and sample results are compared to raw data records. Secondary review must be documented with the signature/initials of the reviewer and the date the review was performed. If any problems are found, sample results must be re-checked. All errors noted or corrections made shall be documented and communicated to the primary reviewer.

At a minimum, the review must ensure the following:

- Sample preparation and analysis information is correct and complete.
- Calibration data is appropriate, accurate and documented.
- Analytical results are correct and complete
- QC samples are within established control limits or otherwise qualified.
- Qualitative identification is accurate.
- Quantitative results including calculations performed are correct.
- Manual integrations are appropriate and documented.
- Dilution factors are correctly recorded and applied.
- Data transfers are verified and correct.
- Non-conformances have been properly documented and communicated.
- The appropriate SOPs and client requirements were followed.
- Internal COC documentation is correct and complete, when required.
- Documentation is complete.

Completeness Review (Administrative Review)

The completeness review is performed by the Project Manager and includes a total overview of the data report to ensure its consistency with project specific requirements. Any errors noted during this review shall be noted and corrected prior to submission of the report to the client.

Detailed procedures for data review are further described in laboratory SOP LP-LB-0003 *Data Review.*

QA Review

QA review may be performed on a percentage of data produced by the laboratory using the internal audit procedures described in Section 5.16.

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13. Data Reports

The results of analyses performed by the must be reported accurately, clearly, unambiguously and objectively in data package reports that include the information necessary to interpret results. The laboratory offers a variety of hardcopy and electronic deliverable (EDD) report formats including a comprehensive data package that contains sufficient information to perform third party validation. The laboratory recognizes that different programs have distinct report formats. The exact type of deliverables required for each project should be based on the intended use of the data and specified during project initiation.

Regardless of report format, each data report must include the following information:

- Title of the Report
- Laboratory name, location and contact information (point of contact, phone and fax number)
- Client Name, project name and/or unique project report number
- Total Number of Pages
- Field Sample ID
- Laboratory Sample ID
- Sample Matrix
- Date of sample collection, receipt, preparation, and analysis.
- Analysis time when holding time is less than 48 hours
- Test Method(s) Used
- Analytical Results, any failures, reporting units and identification of data reported on a dry weight or wet weight basis
- Data Qualifier Definitions
- Project Narrative

Each test report should also include the following information when relevant or necessary:

- Reference to sampling plan where relevant to the validity or application of test results
- A statement to the effect that results relate only to the samples reported
- A statement that the certificate may not be reproduced except in full without the written approval of the laboratory
- A certification statement that the test results meet all requirements of the NELAC standard along with a discussion that provides justification for results that do not meet those requirements.
- A statement of estimated uncertainty
- Opinions and interpretations

The project narrative included with each data report includes a table that summarizes the samples received and provides a correlation between the field sample Ids to the sample ID assigned by the laboratory along with a description of any thing that occurred during sample

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receipt, preparation or analysis that could affect the sample results, including holding time violations, calibration and QC failures and associated corrective actions. Any departures from standard operating procedures or project specifications are also be noted.

Hardcopy data reports from external subcontract shall not be altered and must be included in original form in the final data report provided by STL Burlington. However, data from subcontractors' reports may be added to an electronic deliverable as required to meet the EDD format specified. Subcontracted data shall be clearly identified as such with the name, address, and telephone number for the laboratory performing the test is included in the project report. The originating laboratory (STL Burlington) shall ensure that the test report provided by the subcontract laboratory complies with any relevant project specifications for report format and content.

The release of data reports is authorized by the Laboratory Director or his designee and is documented by his signature and title on the project narrative or with an equivalent electronic identification of the person(s) accepting responsibility for the content of the report.

Where amendments to data reports are required after issue, these shall be in the form of a separate document and/or electronic data deliverable. The revised report must be clearly identified as revised with the date of revision and the initials of the person making the revision. Specific pages of a project report may be revised using the above procedure with an accompanying cover letter indicating the page numbers of the project revised. The original version of the project report must be kept intact and the revisions and cover letter included in the project files.

Electronic Data Deliverables, (EDDs) may be provided in addition to or in lieu of hard copy reports. The laboratory offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, XML, and Text Files. EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process in Section 4.4.1. Once the facility has committed to providing diskettes in a specific format, the coding of the format is performed, documented and validated. The validation of the code is retained as a QC record.

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14. Table 1: STL Facility Locations & Contact Information

STL Laboratory	Location	Phone Number
AEL	Phoenix, AZ	602-437-3340
STL Mobile	Mobile, AL	251-666-6633
STL Los Angeles	Santa Ana, CA	714-258-8610
STL Sacramento	West Sacramento, CA	916-373-5600
STL San Francisco	Pleasanton, CA	925-484-1919
STL Denver	Arvada, CO	303-736-0100
STL Connecticut	Shelton, CT	203-929-8140
STL Pensacola	Pensacola, FL	850-474-1001
STL Tallahassee	Tallahassee, FL	850-878-3994
STL Tampa	Tampa, FL	813-885-7427
STL Savannah	Savannah, GA	912-354-7858
STL Chicago	University Park, IL	708-534-5200
STL Valparaiso	Valparaiso, IN	219-464-2389
STL New Orleans	Kenner, LA	504-469-3685
STL Westfeld	Westfield, MA	413-572-4000
STL St. Louis	Earth City, MO	314-298-8566
STL Edison	Edison, NJ	732-549-3900
STL Buffalo	Amherst, NY	716-691-2600
STL Newburgh	Newburgh, NY	845-562-0890
STL North Canton	North Canton, OH	330-497-9396
STL Pittsburgh	Pittsburgh, PA	412-963-7058
STL Knoxville	Knoxville, TN	865-291-3000
STL Austin	Austin, TX	512-244-0855
STL Corpus Christi	Corpus Christi, TX	361-289-8673
STL Houston	Houston, TX	713-690-4444
STL Richland	Richland, WA	509-375-3131
STL Seattle	Tacoma, WA	253-922-2310
STL Burlington	Colchester, VT	802-655-1203

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15. Table 2: Analytical Capabilities

Test Method	Description
ASTM D1946	Fixed Gases in Air
ASTM D4318	Liquid Limit, Plastic Limit, Plasticity
ASTM D854	Specific Gravity
ASTM D2216	Water Content of Soil and Rock
ASTM D2217	Wet Preparation Technique for Particle Size Analysis
ASTM D421	Dry Preparation Technique for Particle Size Analysis
ASTM D422	Particle Size Analysis
ASTM D5084	Hydraulic Conductivity
ASTM D2434	Permeability of Granular Soils
ASTM D2937	Density in Soils / Drive Cylinder Method
ASTM D2974	% Moisture, % Ash, % Organic Matter
ASTM D698	Compaction Characteristics of Soil using Standard Effort
ASTM D1140	Material in Soil Finer than #200 Sieve (75 um)
ASTM D2487	Classification of Soils for Engineering Purposes
NA	Long Tube Column Settling Test
EPA	Total Suspended Particulate Matter in the Atmosphere
EPA 504.1	Microextractables / EDB, DBCP, 1,2,3-TCP
EPA 524.2	Volatile Organic Compounds by GC/MS
EPA 1624	Volatile Organic Compounds by GC/MS, Isotope Dilution
EPA 1625	Semivolatile Organic Compounds by GC/MS, Isotope Dilution
EPA 3C	Permanent Gas Analysis
EPA 25C	Non-Methane Organic Compounds
EPA TO4	Pesticides / PCBs in Ambient Air
EPA TO13	Semivolatile Organic Compounds in Ambient Air by GC/MS
EPA TO14A	Volatile Organic Compounds in Ambient Air by GC/MS
EPA TO15	Volatile Organic Compounds in Ambient Air by GC/MS
EPA 8015M	Diesel Range Organics
EPA 8015M	Gasoline Range Organics
EPA 8260	Volatile Organic Compounds by GC/MS
EPA 8270	Semivolatile Organic Compounds by GC/MS
EPA 8081	Organochlorine Pesticides by GC/ECD
EPA 8082	Polychlorinated Biphenyl Aroclors
EPA 8082	Polychlorinated Biphenyl Congeners
EPA 8330	Nitroaromatics & Nitramines by HPLC
EPA 8151	Herbicides
EPA 5030	Purge and Trap
EPA 5035	Closed System Purge and Trap
EPA 3510	Separatory Funnel Extraction
EPA 3520	Continuous Liquid-Liquid Extraction
EPA 3540	Soxhlet Extraction
EPA 3541	Automated Soxhlet Extraction
EPA 3550	Ultrasonic Extraction
EPA 3580	Waste Dilution

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Table 2: Analytical Capabilities continued

Test Method	Description
EPA 3005	Acid Digestion of Waters
EPA 3010	Acid Digestion of Waters
EPA 3050	Acid Digestion of Soils
EPA 6010B	Metals by ICP-AES
EPA 6020	Metals by ICP-MS
EPA 200.7	Metals by ICP-AES
EPA 200.8	Metals by ICP-MS (Drinking Water Only)
EPA 7196A	Hexavalent Chromium (Water Only)
EPA 7470 / 7471	Mercury CVAA
EPA 9040 / 9045	pH
EPA 1311	Toxicity Characteristic Leachate Procedure (TCLP)
EPA 1312	Synthetic Precipitate Leachate Procedure (SPLP)
Status & Trends	Alkyltins
Status & Trends	SIM Polyaromatic Hydrocarbons (PAH)
EPA 245.1	Mercury CVAA
EPA 9081	Cation Exchange Capacity
EPA 1010	Ignitability
Status & Trends	Pesticides and PCBs
EPA 310.1	Alkalinity
SM2320B	Alkalinity
EPA 350.2	Ammonia
AVS/SEM	Acid Volatile Sulfide / Simultaneously Extracted Metals
EPA 405.1	Biochemical Oxygen Demand
EPA 300.0 / 9056	Inorganic Anions (except Nitrite)
SM5210B	CBOD
EPA 410.4	Chemical Oxygen Demand
EPA 325.2 / 9250	Chloride
EPA 335.3 / 335.4	Cyanide, Total
EPA 9012	Cyanide, Total
EPA 9012	Cyanide, Amenable to Chlorination
EPA 360.2	Dissolved Oxygen
SM3500 Fe D	Ferrous Iron
EPA 340.2	Fluoride
EPA 9095	Free Liquid
EPA 130.2	Hardness, Total
EPA 200.7	Hardness, Total
EPA 351.3	Kjeldahl Nitrogen
EPA 353.2	Nitrate-nitrite
EPA 354.1	Nitrite
EPA 350.2	Organic Nitrogen
EPA 365.2	Orthophosphate
EPA 150.1	pH

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Table 2: Analytical Capabilities continued

Test Method	Description
EPA 420.1 / 9065	Phenols
EPA 365.2	Phosphorus, Total
SM2520B	Salinity
EPA 160.1	Total Dissolved Solids
EPA 160.2	Total Suspended Solids
EPA 160.3	Total Solids
EPA 160.4	Total Volatile Solids
EPA 160.5	Settable Solids
EPA 120.1 / 9050A / SM2510B	Specific Conductance
EPA 375.4 / 9038	Sulfate
EPA 415.1 / 9060	Total Organic Carbon (Water Only)
Lloyd Kahn TOC	Total Organic Carbon (Soil Only)
EPA 180.1	Turbidity
CLP ILM04.1	Metals by ICP-AES, Cyanide, and Mercury by CVAA
CLP ILM05.3	Metals by ICP-AES, ICP-MS, Cyanide, and Mercury by CVAA
CLP OLC02.1	Volatiles by GC/MS
CLP OLC02.1	Semivolatiles by GC/MS
CLP OLC02.1	Pesticides / PCBs by GC/ECD
CLP OLM04.3	Volatiles by GC/MS
CLP OLM04.3	Semivolatiles by GC/MS
CLP OLM04.3	Pesticides / PCBs by GC/ECD
CLP SOM01.0	Volatiles by GC/MS
CLP SOM01.0	Semivolatiles by GC/MS
CLP SOM01.0	Pesticides / PCBs by GC/ECD
EPA 3620B	Florisil Cleanup
EPA 3630C	Silica Gel Cleanup
EPA 3640A	Gel Permeation Chromatography
EPA 3660B	Sulfur Cleanup
EPA 3665A	Sulfuric Acid Cleanup

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16. Table 3: Certification List

Program	Certification Type	Certificate Number
Arizona	State	AZ0665
Connecticut	State	PH-0751
Delaware DNREC	State	NA
Florida	Secondary NELAC	E87467
Maine	State	VT0008
Massachusetts	State	M-VT0008
NFESC[1]	Agency Approval	NA
New Hampshire	Secondary NELAC	200604
New Jersey	Primary NELAC	VT972
New York	Secondary NELAC	10391
Pennsylvania	Secondary NELAC	489
Rhode Island	State	81
USACE[2]	Agency Approval	NA
Vermont	State	VT4000

[1] Naval Facilities Engineering Service Center

[2] US Army Corps of Engineers

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17. Table 4: Major Equipment Listing

Instrument Type	Manufacturer	Model	Purchase Date	Autosampler
ICP-AES	Thermo Jarrell Ash (4) S/N 234390	61E Trace	1994	Yes
	Thermo Jarrell Ash (5) S/N 236390	61E Trace	1996	Yes
	Thermo Jarrell Ash (6) S/N 435690	61E Trace	1997	Yes
ICP-MS	Thermo Elemental S/N X0288	X5	2003	Yes
Mercury Analyzer	Leeman Labs S/N 112-00064-1	PS200	1991	Yes
	Leeman Labs S/N 2031	Hydra AA	2003	Yes
GC/MS Semivolatiles	Hewlett-Packard (P) GC S/N 3203A40985 MS S/N 3188A03495	5890II – GC 5971 - MS	1998	Yes
	Hewlett-Packard (Q) GC S/N 3203A40983 MS S/N 3188A03498	5890II – GC 5971 - MS	1998	Yes
	Hewlett-Packard (R) GC S/N 3203A40984 MS S/N 3188A03506	5890II – GC 5971 - MS	1998	Yes
	Hewlett-Packard (Y) GC S/N 3336A60147 MS S/N 3524A2777	5890II – GC 5972 - MS	1998	Yes
	Hewlett-Packard (U) GC S/N 3336A61525 MS S/N 3549A03238	5890II – GC 5972 - MS		Yes
	Hewlett-Packard (Z) GC S/N US00036343 MS S/N US02440321	6890 – GC 5973 - MS	2000	Yes
GC/MS Volatiles	Hewlett-Packard (L) GC S/N 3203A40982 MS S/N 3188A03410	5890II – GC 5971 – MS	1998	Yes
	Hewlett-Packard (M) GC S/N 3203A40980 MS S/N 3188A03486	5890II – GC 5971 – MS	1998	Yes
	Hewlett-Packard (N) GC S/N 3203A40979 MS S/N 418803507	5890II - GC 5971 - MS	1998	Yes

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Instrument Type	Manufacturer	Model	Purchase Date	Autosampler
	Hewlett-Packard (O) GC S/N 3203A40981 MS S/N 3188A03504	5890II – GC 5971 – MS	1998	Yes
Ion Chromatograph	Dionex S/N	DX-500	1995	Yes
	Dionex S/N	ICS-1000	2004	Yes
TOC	Carlo Erba S/N 249146	EA-1108	1991	Yes
	Carlo Erba S/N 220465	EA-1108	1991	Yes
	Shimadzu S/N 63890260-22	TOC-5000A	1997	Yes
TKN Digestion System	Tecator S/N	1015	1991	No
UV/VIS	Genesys S/N 35GB029021	Spectronic 20	1999	No
	Genesys S/N 35GE165024	Spectronic 20	2002	No
	Lachat S/N	Quick Chem 8000	2003	Yes
	Lachat S/N A83000-591	Quick Chem 8000	2000	Yes
Autotitrator	Man-Tech	Titra-Sip	2002	Yes
pH Meter	Beckman S/N 166928	45	1991	No
Dissolved Oxygen Meter	YSI S/N 95A28272	58	1995	No
	YSI S/N 95A28273	58	1995	No
BOD Assay	Man-Tech			
Turbidimeter	HF Scientific S/N 208463	Micro 100	2001	No
Automated Distillation Apparatus	Westco S/N 1090	Easy Dist	2002	No
	Westco S/N 1091	Easy Dist	2002	
	Labconco S/N	Rapid Still II	1992	

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Instrument Type	Manufacturer	Model	Purchase Date	Autosampler
	Labconco S/N 232698	Rapid Still II	1993	
	Labconco S/N 000693491E	Rapid Still II	1999	
Midi Distillation Apparatus	Lab Crest S/N A9R0409	110-10-R	2000	No
	Lab Crest S/N C9P0109	110-4-R	2000	No
	Lab Crest S/N A7Y0209	110-10-R	2000	No
	Lab Crest S/N D0R0200	110-4-R	2000	No
COD	HACH S/N 11000022452			No
GPC	J2 Scientific (I) S/N 02D-1030-2.1	Autoprep 1000	2002	Yes
GPC	J2 Scientific (H) S/N 02D-1031-2.1	Autoprep 1000	2001	Yes
GPC	J2 Scientific (J) S/N 03G1076-3.0	Autoprep 1000	2003	Yes
GPC	Analytical Biochemistry S/N 9137 SI	1000		
GC Semivolatiles	Hewlett-Packard (2865) S/N 3203A41769	5890II Dual ECD		Yes
	Hewlett-Packard (4237) S/N US104314	6890 Dual PFPD	2004	Yes
	Hewlett-Packard (7424) S/N US10332093	6890 Dual ECD	2003	
	Hewlett-Packard (2087) S/N 3115A34941	5890II Dual ECD	1991	Yes
	Hewlett-Packard (3327) S/N 3336A59080	5890II Dual ECD	1998	Yes
	Hewlett-Packard (2620) S/N 3203A41056	5890II Dual ECD	1998	Yes
	Hewlett-Packard (2404) S/N 3133A37364	5890II Dual ECD	1991	Yes
	Hewlett-Packard (2618) S/N 3203A41055	5890II Dual ECD	1987	Yes
	Hewlett-Packard (2624) S/N 3203A41057	5890II Dual ECD	1998	Yes

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Instrument Type	Manufacturer	Model	Purchase Date	Autosampler
	Hewlett-Packard (2860) S/N 2950A27078	5890 Dual FPD	1990	Yes
	Hewlett-Packard (2622) S/N 3203A41058	5890II Dual FPD	1987	Yes
	Hewlett-Packard (3012) S/N 3235A45259	5890II Dual FID	1984	Yes
	Hewlett-Packard (3328) S/N 3336A58806	5890II Dual FID	1998	Yes
	Hewlett-Packard (0825) S/N US10202136	6890II Dual ECD	2002	Yes
	Hewlett-Packard (0911) S/N US10230082	6890II Dual ECD	2002	Yes
GC Volatiles	Hewlett-Packard (0869) S/N 2443A04359	5890A PID / FID	1996	Yes
	Hewlett-Packard S/N 3133A37550	5890	1996	Yes
LC	Waters (1208) S/N 04790RP	600	1988	Yes
	Waters (1488) S/N 6PLEPF138	600E	1991	Yes
	Waters (3330) S/N 415M	600	1997	Yes
	Waters (0601) S/N 6PLEPE169	600E		
	Waters (1) S/N MX6E5194M	996 PDA		No
	Waters (2) S/N MX5PM93960	996 PDA		No
Air	Hewlett Packard (V) S/N 3549A03239	5971	1998	Yes
	Hewlett Packard (B) S/N US30965342	5973	2003	Yes
	Hewlett Packard (E) S/N US44621242	5973	2005	Yes
	Hewlett Packard (F) S/N US52420622	5973	2005	Yes
	Varian S/N 1467	3600	1998	Yes

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18. Table 5: QC Summary, Purpose and Recommended Frequency

Lab QC	Purpose	Recommended Frequency
Method Blank	To assess background interference or contamination.	1 per batch of 20 or less samples
Laboratory Control Sample	To assess the laboratory's ability to recover the target analytes from a clean matrix	1 per batch of 20 or less samples
Field QC	**Purpose**	**Recommended Frequency**
Duplicate Sample	To provide information on the heterogeneity of the sample matrix or to determine the precision of the method for a specific matrix	1 per 20 project samples
Matrix Spike	To assess the performance of the method as applied to a specific site matrix; to determine the effect of the matrix on a method's recovery efficiency; to determine the bias of the method in the specific sample matrix.	1 per 20 project samples
Matrix Spike Duplicate	Same as matrix spike; to provide information on the homogeneity of the matrix; to determine the precision of the method for a specific matrix.	1 per 20 project samples
Trip Blank (VOA Only)	To assess for contamination that may occur during shipment or transport to the laboratory	1 per sampling event
Equipment Blank	To assess the effectiveness of the procedure used to decontaminate sampling equipment.	Per Project DQOs
Field Blank	To assess the contribution of interference or contamination that may occur during a sampling activity or transport to the laboratory.	Per Project DQOs
Field Duplicate	To measure the representativeness of sampling and the effect of site matrix on precision.	Per Project DQOs

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19. APPENDIX A: REFERENCES

EPA Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality Related Documents, EPA QA/G-6, US EPA, Office of Environmental Information, March 2001.

EPA Requirements For Quality Management Plans, EPA QA/R-2, US EPA, Office of Environmental Information, March 2001.

EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, US EPA, Office of Environmental Information, March 2001.

EPA Quality Manual for Environmental Programs, 5360 A1, US EPA, Office of Environmental Information, March 2001.

General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025, December 1999.

Good Automated Laboratory Practices, EPA 2185, 1995.

Quality Assurance Project Plan, HQ Air Force Center for Environmental Excellence, Version 3.1, August 2001.

National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards, EPA600/R-98/151, USEPA Office of Research and Development, July 2002.

Navy Installation Restoration Laboratory Quality Assurance Guide, Interim Guidance Document, Naval Facilities Engineering Service Center, February 1996.

Navy Installation Restoration Chemical Data Quality Manual, Navy IR CDQM, September 1999.

Quality Systems Manual for Environmental Laboratories, Department of Defense, Version 2, June 2002.

Shell for Analytical Chemistry Requirements, US Army Corps of Engineers, December 1998.

STL Quality Management Plan, M-Q-001 Revision 6, Severn Trent Laboratories, Inc. December 2003.

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20. APPENDIX B: TERMS & DEFINITIONS

Accuracy: the degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Audit: a systematic evaluation to determine the conformance to specifications of an operational function or activity.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above-mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Chain of Custody (COC): an unbroken trail of accountability that ensures the physical security of samples, data and records.

Compromised Sample: a sample received in a condition that jeopardizes the integrity of the results.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Equipment Blank: a portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Rinsate.

Document Control: the act of ensuring that documents (electronic or hardcopy and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Field Blank: a blank matrix brought to the field and exposed to field environmental conditions.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

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Internal Chain of Custody: an unbroken trail of accountability that ensures the physical security of samples, data and records. Internal Chain of Custody refers to additional documentation procedures implemented within the laboratory that includes special sample storage requirements, and documentation of all signatures and/or initials, dates, and times of personnel handling specific samples or sample aliquots.

Instrument Detection Limit (IDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Laboratory Quality Manual (LQM): a document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system.

Matrix: the substrate of a test sample. Common matrix descriptions are defined in the following table:

Matrix	Description
Air	Air samples as analyzed directly or as adsorbed into a solution or absorption matrix and desorbed.
Aqueous	Aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine source. Includes surface water, groundwater and effluents.
Drinking Water	Aqueous sample that has been designated a potable water source.
Saline	Aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.
Liquid	Liquid with <15% settleable solids.
Solid	Soil, sediment, sludge or other matrices with ≥15% settleable solids.
Waste	A product or by-product of an industrial process that results in a matrix not previously defined.
Tissue	Sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

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Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Precision: an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

Proficiency Testing: determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.

Proprietary: belonging to a private person or company.

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 Assurance Project Plan (QAPP): a formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

Quality Control (QC): the overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control Sample: a control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Quality Management Plan (QMP): a formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory to ensure the quality of its product and the utility of the product to its users.

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Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA/QC.

Quantitation Limit (QL): the minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Raw Data: any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and 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/optical media, including dictated observations, and recorded data from automated instruments. Reports specifying inclusion of “raw data” do not need all of the above included, but sufficient information to create the reported data.

Record Retention: the systematic collection, indexing and storing of documented information under secure conditions.

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Sampling and Analysis Plan (SAP): a formal document describing the detailed sampling and analysis procedures for a specific project.

Selectivity: the capability of a measurement system to respond to a target substance or constituent.

Sensitivity: the difference in the amount or concentration of a substance that corresponds to the smallest difference in a response in a measurement system using a certain probability level.

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Storage Blank: a blank matrix stored with field samples of a similar matrix.

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Systems Audit: a thorough, systematic, on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

Test Method: defined technical procedure for performing a test.

Traceability: the property of a result of a measurement that can be related to appropriate international or national standards through an unbroken chain of comparisons.

Trip Blank: a blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Verification: confirmation by examination and provision of evidence against specified requirements.

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21. Appendix C: Sample Container Guide

Matrix	Parameter	Test Method[1]	Container[2]	Container Size	Preservative	Holding Time
Air	Permanent Gases	3C	Summa Can, Tedlar Bag	1 or 6 L / 1 Bag	NA	30 Day / 72 Hour
Air	Non Methane Organic Compounds	25C	Summa Can, Tedlar Bag	1 or 6 L / 1 Bag	NA	30 Day / 72 Hour
Air	Volatile Organic Compounds	TO14 & TO15	Summa Can, Tedlar Bag	1 or 6 L / 1 Bag	NA	30 Day / 72 Hour
Air	SVOA	TO13	PUF	3 inch	NA	7 Day
Air	PCB	TO4	PUF	3 inch	NA	7 Day
Soil	Geotechnical	ASTM	G	250 - 500 mL	NA	NA
Soil	Metals	SW-846	G	125 - 500 mL	4°C ±2	6 Months
Soil	Organic Extractables	SW-846	G	125 - 500 mL	4°C ±2	14 Days
Soil	General Chemistry	Various	G	125 - 500 mL	4°C ±2	Various
Water	Alkalinity	310.1	P	1 Liter	4°C ±2	14 Days
Water	Ammonia	350.2	P	1 Liter	4°C ±2 & H_2SO_4 pH < 2	28 Days
Water	Inorganic Anions	300.0	P	1 Liter	4°C ±2	28 Days / 48 Hr
Water	Perchlorate	314.0 & 331.0	P	250 mL	4°C ±2	28 Days
Water	BOD	405.1	P	1 Liter	4°C ±2	48 Hour
Water	COD	410.4	P	250 mL	4°C ±2 & H_2SO_4 pH < 2	28 Days
Water	Chloride	300.0 & 9056, 325.2 & 9250	P	1 Liter	4°C ±2	28 Days
Water	Chromium IV	7196A	P	1 Liter	4°C ±2	24 Hour
Water	Color	110.1	P	500 - 1000 mL	4°C ±2	48 Hour
Water	Cyanide	335.2, 335.3, 335.4, 9012	P	250 mL	4°C ±2, ascorbic acid & NaOH pH >12	14 Days
Water	Fluoride	300.0	P	1 Liter	4°C ±2	28 Days
Water	Hardness (Titration)	130.2	P	500 mL	4°C ±2 & HNO_3 pH < 2	6 Months
Water	Metals	200.7, 200.8, 6010, 6020	P	500 mL	4°C ±2 & HNO_3 pH < 2	6 Months
Water	Mercury	245.1, 7470	P	500 mL	4°C ±2 & HNO_3 pH < 2	28 Days
Water	Nitrogen, Kjeldahl (TKN)	351.3	P	1 Liter	4°C ±2 & H_2SO_4 pH < 2	28 Days
Water	Nitrate as N	353.2	P	250 mL	4°C ±2 & H_2SO_4 pH < 2	28 Days
Water	Nitrite	354.1	P	250 - 1000 mL	4°C ±2	48 Hour
Water	Phenols	420.1	G	1 Liter Amber	4°C ±2 & H_2SO_4 pH < 2	28 Days
Water	Phosphorus, Total	365.2	P	250 – 1000 ml	4°C ±2 & H_2SO_4 pH < 2	28 Days

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Sample Container Guide Continued

Matrix	Parameter	Test Method[1]	Container[2]	Container Size	Preservative	Holding Time
Water	Orthophosphate	365.2	P	1 Liter	4°C ±2	48 Hour
Water	pH	150.1 & 9040	P	500 mL	None	Immediately
Water	Solids, Dissolved (TDS)	160.1	P	1 Liter	4°C ±2	7 Days
Water	Solids, Suspended (TSS)	160.2	P	1 Liter	4°C ±2	7 Days
Water	Solids, Total (TS)	160.3	P	1 Liter	4°C ±2	7 Days
Water	Solids, Volatile (TVS)	160.4	P	1 Liter	4°C ±2	7 Days
Water	Settable Solids	160.5	P	1 Liter	4°C ±2	7 Days
Water	Specific Conductance	120.1 & 9050	P	1 Liter	4°C ±2	28 Days
Water	Sulfate	300.0 & 9056, 375.4 & 9038	P	1 Liter	4°C ±2	28 Days
Water	Sulfide	376.2, 9030/9034	P	500 mL	4°C ±2 & zinc acetate & NaOH pH >9	7 Days
Water	Total Organic Carbon (TOC)	415.1 & 9060	G-TLS	40 mL	4°C ±2 & H_2SO_4 pH < 2	28 Days
Water	Turbidity	180.1	P	1 Liter	4°C ±2	28 Days
Water	Diesel Range Organics (DRO)	8015M	G-TLC	1 Liter	4°C ±2	7 / 40 Days
Water	Gasoline Range Organics (GRO)	8015M	G-TLS	40 mL	4°C ±2, HCl pH <2	14 Days
Water	Volatile Organic Compounds	524.2 & 8260, 1624	G-TLS	40 mL	4°C ±2, HCl pH <2	14 Days
Water	Pesticides & PCBs	8081 & 8082	G-TLC	1 Liter Amber	4°C ±2	7 / 40 Days
Water	Herbicides	8151	G-TLC	1 Liter Amber	4°C ±2	7 / 40 Days
Water	SVOA & PAHs	8270, 1625	G-TLC	1 Liter Amber	4°C ±2	7 / 40 Days
Water	Microextractables	504.1	G-TLS	40 mL	Sodium Thiosulate	14 Day
Water	Dissolved Gases	RSK-175	G-TLS	40 mL	CO_2-unpreserved / HCl	14 Day
Water	Explosives	8330	G-TLC	1 Liter Amber	4°C ±2	14 Day
Water	CBOD	SM5210B	P	1 Liter	4°C ±2	48 Hour

[1] References: 40CFR Part 136, Methods for the Chemical Analysis of Water and Wastes, SW-846 Test Methods for Evaluating Solid Waste, ASTM.

[2] Acronym Definitions: P = Polyethylene G = Glass G-TLC = Glass with Teflon® lined caps G-TLS = Glass with Teflon® lined septum

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Attachment 1-3

STL PCB SOPs

STL BURLINGTON
SOP CHANGE-IN-PROGRESS ATTACHMENT (CIPA)

SOP Title: POLYCHLORINATED BIPHENYLS (PCB'S) BY GC/ECD SW-846 METHOD 8082
SOP No: LM-GC-8082
Revision: **7**
Date Effective: 12/12/05
CIPA Date Effective: 07/27/06

Approval Signatures:

QA Manager: Kirstin McCracken **Date:** July 27, 2006

Organic Manager: Jacob L. Bailey **Date:** July 27, 2006

The following revisions or additions in BOLD TEXT were made to the referenced SOP. These changes were implemented on the CIPA Date Effective indicated above.

Page 7: Section 10.5 ICV – Second Source Standard

After each calibration, verify the accuracy of the initial calibration by analyzing the ICV (Appendix A). The calculated concentration of each analyte must be within ± ~~**15%**~~ **20%** of the theoretical concentration. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate. The acceptance criteria must be met on both columns for single component pesticides.

Page 16: Revise Table 4: The revisions are shown on the next page.

SOP CHANGE-IN-PROGRESS ATTACHMENT (CIPA)
SOP No: LM-GC-8082 r7

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CF: RSD ≤ 20% Linear Regression: r ≥ 0.99 (0.995 for DoD)	Correct problem, reanalyze, repeat calibration.
ICV	After each initial calibration	%Difference ± ~~15%~~ **20%** from expected value	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±15%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and bracketed samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	< RL DoD: ≤ ½ RL If analyte in any sample ≥ RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	Evaluated against control limits in Table 1	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch SD: Per client request	Evaluated against control limits in Table 1	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate Spike	All field and QC samples	Evaluated against control limits in Table 1	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

STL BURLINGTON
SOP CHANGE -IN-PROGRESS ATTACHMENT (CIPA)

SOP Title: Polychlorinated Biphenyls (PCBs) by GC/ ECD SW-846 8082
SOP No: LM-GC-8082
Revision: 7
Date Effective: 12/12/05
CIPA Date Effective: 01/20/06

Approval Signatures:

QA Manager: Kirstin McCracken **Date:** January 20, 2006

Organic Manager: Jennifer L. Clements **Date:** January 20, 2006

The following revisions or additions in BOLD TEXT were made to the referenced SOP. These changes were implemented on the CIPA Date Effective indicated above.

Table 1: Target Analyte List, Reporting Limits[1], and Control Limits[2] as Accuracy (%R) and Precision[3] (RPD)

Analyte	Reporting Limit[1]		Water		Solid, Chemical Material, Tissue	
	Water ug/L	Soil ug/Kg	%R	RPD[4]	%R	RPD[3]
AR1016	0.50	17	55-125	≤ 35	55-120	≤ 30
AR1221	0.50	17	NA	NA	NA	NA
AR1232	0.50	17	NA	NA	NA	NA
AR1242	0.50	17	NA	NA	NA	NA
AR1248	0.50	17	NA	NA	NA	NA
AR1254	0.50	17	NA	NA	NA	NA
AR1260	0.50	17	55-120	≤ 30	60-130	≤ 35
DCB (Surrogate)	NA	NA	55-120	NA	60-125	NA
TCMX[4] (Advisory Surrogate)	**NA**	**NA**	**30-150**	**NA**	**30-150**	**NA**

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.
[2] The in-house statistical control limits posted in this table are those in effect on the revision date of this SOP. These limits are subject to change based on performance trends.
[3] RPD for MS/MSD only.
[4] The control limits for TCMX are advisory. Corrective action is not required if the recovery exceeds the upper or lower control limit.

STANDARD OPERATING PROCEDURE
POLYCHLORINATED BIPHENYLS (PCB'S) BY GC/ECD
SW-846 METHOD 8082
Applicable Matrices: Non-Potable Water, Solid and Chemical Materials
Standard Compound List and Reporting Limits: See Table 1

APPROVAL SIGNATURES

Laboratory Director: _____ Date: December 12, 2005
Christopher A. Ouellette

QA Manager: _____ Date: December 12, 2005
Kirstin L. McCracken

Organics Manager: _____ Date: December 12, 2005
Jennifer L. Clements

Proprietary Information Statement:

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1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the determination of concentrations of Polychlorinated Biphenyls (PCBs) in extracts derived from non-potable water, solids, tissue, air, and chemical materials including TCLP leachates, using dual column Gas Chromatography with Electron Capture Detectors (GC/ECD). This SOP is applicable to the analytical procedure only; the extraction and extract cleanup methods referenced in this SOP are described in the following laboratory SOPs:

LM-OP-3510	Separatory Funnel Extraction
LM-OP-3540	Soxhlet Extraction
LM-OP-3541	Automated Soxhlet Extraction
LM-OP-3550	Ultrasonic Extraction
LM-OP-Cleanup	Extract Cleanup Procedures
LM-OP-GPC	Gel Permeation Chromatography (GPC)

1.2 The analytes that can be determined by this procedure and their associated Reporting Limits (RL) are listed in Table 1, Section 18.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using an appropriate matrix-specific extraction technique. After extraction, the extract may be subject to cleanup depending on the nature of sample matrix and the target analytes. After cleanup, the extract is analyzed by injecting a 2-µl aliquot into a dual capillary column GC/ECD.

2.2 This procedure is based on SW-846 Method 8082, Revision 0, December 1996.

3.0 DEFINITIONS

A list of terms and definitions is given in Appendix C.

4.0 INTERFERENCES

4.1 Contaminated solvents, reagents or equipment can cause interferences. To reduce the occurrence of this type of interference, glassware must be cleaned thoroughly before use following the procedure given in laboratory SOP LM-OP-Glass *Glassware Washing*. Solvents and acids are lot- tested and approved for use before delivery to the laboratory in accordance with STL-T-001 *Testing of Solvent and Acids*. Solvents should be stored in an area away from organochlorine compounds to minimize contamination.

4.2 Phthalate esters introduced during sample preparation can pose a problem in the determination of pesticides. Common flexible plastics contain varying amounts of phthalate esters, and these can be easily extracted or leached during extraction. To minimize this interference, avoid contact with any plastic materials.

4.3 Non-target compounds co-extracted from the sample matrix can also cause interference, the extent of which will vary considerably depending on the nature of the samples. Elemental sulfur is often found in sediment samples and its presence will result in broad peaks. Samples are screened before analysis and those samples that contain high levels of sulfur are subject to cleanup using activated copper before analysis (SW-846 3660B). Waxes, lipids, other high molecular weight materials and co-eluting organophosphorous pesticides may be removed by extract cleanup with GPC (SW-846-3640A). Co-eluting chlorophenols can be eliminated by cleanup with silica gel (SW-846 3630C), or Florisil (SW-846 3620B), or Sulfuric acid Cleanup (SW-846 3665A) may be used to eliminate certain organochlorine pesticides and elevated baselines.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 Specific Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 Primary Materials Used

Table 2, Section 18 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. Note: The table does not include all materials used in the procedure. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used can be found in Section 7. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 EQUIPMENT AND SUPPLIES

6.1 Autosampler Vials, National Scientific or equivalent.

6.2 Computer Hardware/Software: GC Acquisition Platform - VAX 4505 (GVAX) Multichrom V2.11. Data Processing - Hewlett-Packard 9000-series computers, an HP9000 D250 (Chemsvr4) and an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5.

6.3 GC/ECD: with dual columns, dual ECDs, and auto-sampler capable of a 2-µl injection split onto two columns: HP 5890 with Leap Technology CTC A200SE and A200S Fisons autosamplers, or equivalent.

6.4 GC Columns: A dual fused silica capillary column system that will provide simultaneous primary and confirmation analyses.

- RTX-5, (30m x 0.25 mmID x 0.25um)
- RTX-35, (30m x 0.25 mmID x 0.25um)
- Equivalent columns may be used, provided the elution orders are documented and compound separations are maintained.

6.5 Hydrogen Generator: Whatman.

6.6 Volumetric Syringes, Class "A" (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

- Hexane JT Baker Ultra-Resi analyzed.

7.2 Standards

Stock standard solutions are purchased made from commercial vendors. Intermediate and working standards solutions are prepared in the laboratory by diluting a known volume of stock standard in an appropriate solvent and diluting to a specified volume. Standard preparation procedures for intermediate and working standard solutions are provided in Appendix A.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 Sample extracts must be stored at 4°C ± 2° until the time of analysis. The analytical holding time is 40 days from date of sample extraction.

8.2 Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 The minimum frequency requirements, acceptance criteria and recommended corrective action for all QC samples are summarized in Section 18, Table 3. Below is a summary of each type of QC sample that is analyzed with the method.

9.2 A Method Blank (MB) and Laboratory Control Sample (LCS) are prepared with each extraction batch. These samples show that the laboratory is in control, independent of the sample matrix.

9.3 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) are prepared with each extraction batch. Project specific MS/MSD are performed per client request. Sample Duplicates (SD) are performed per client request. These samples show the effect of the sample matrix on the accuracy and precision of the method.

9.4 A Surrogate spike is added to all field and QC samples before extraction to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix.

9.5 Instrumental QC standards include a five-point ICAL is generated for Aroclor 1016 and 1260 (referred to as AR1660) and an initial one point calibration for all other Aroclors. After the ICAL, an Initial Calibration Verification (ICV) standard, also referred to as a second source standard, containing Aroclor 1660 is analyzed to verify the ICAL standard formulation. Continuing Calibration Verification (CCV) standards are analyzed before sample analysis, every ten samples thereafter, and at the end of the run to assess instrument drift.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Operating Conditions

Install a five meter deactivated guard column to the injection port and connect the guard column to the separate analytical columns using a glass “Y”. Then attach the analytical columns to the dual ECD detectors.

The recommended instrument operating conditions are as follows:

Initial Temperature:	130°C for 1 minute
Temperature Program:	20°C per minute to 190°C to 5°C per minute to 225°C to 20.0°C per minute to 300°C. Hold for 6 minutes.
Detector Temperature	310°C
Injector Temperature:	225°C
Injection volume:	2μL
Carrier Gas:	Hydrogen (supplied by hydrogen generators)

Optimize the flow rate of the carrier gas by injecting an un-retained substance onto the column at an isothermal oven state and adjusting the flow to obtain the recommended dead volume time.

10.2 Initial Calibration

Before initial or daily calibration, inject an instrument blank (IBLK) consisting of hexane blank to bring the GC/ECD system online.

A multi-point calibration of AR1660 at five concentrations is sufficient in demonstrating linearity. AR1660 includes most of the peaks represented in the other five Aroclors. For the remaining Aroclors, a midlevel standard is analyzed to aid in pattern recognition and is used as a single point calibration standard. Five point calibrations for these aroclors

will only be performed when required by the client or when specified as a regulatory requirement. A minimum of three to five peaks per aroclor is used for quantificaton. The calibration standards are introduced using the same technique that is used for sample extracts described in Section 11.

Inject 2-µl of each calibration standard and calculate the Calibration Factor (CF), mean CF and Percent Relative Standard Deviation (% RSD) for each analyte on both columns (Appendix B). The %RSD for each peak must be ≤ 20 % for the curve to be considered acceptable. If any of the results are outside QC criteria, the cause of the problem is investigated and corrected prior to analysis of samples.

10.3 Alternate Quantificaton. In some cases, it may be preferable to use linear regression to quantify the compounds. The following approaches may be used:

Linear Regression - A curve of concentration vs. peak area is generated for each analyte and the correlation coefficient is calculated. The calibration must have a correlation coefficient (r) ≥ 0.99 (0.995 for DoD) for acquisition of samples to continue. The use of linear regression requires a minimum of 5 calibration points. See SW-846 Method 8000B for linear regression calculations.

Once a method of calibration is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is generated.

10.4 Retention Time Windows

When a new GC column is installed, RT windows are established for 3-5 peaks for Aroclor 1660 by analyzing three standards over a 72-hour period and calculating the mean RT and Standard Deviation (SD). The RT window is calculated as mean RT ± 3SD of the three standards. If the SD is <0.01 minutes, the laboratory may use a default SD of 0.01 minutes.

If, in the professional judgment of the analyst, this results in an RT window that is too tight and would favor false negatives, the laboratory may opt use an alternate method to determine the RT windows as follows: using the RT of the midpoint initial calibration standard, calculate the RT window using ± 0.05 minutes from the midpoint of the RT in the initial calibration.

10.5 ICV – Second Source Standard

After each calibration, verify the accuracy of the initial calibration by analyzing the ICV (Appendix A). The calculated concentration of each analyte must be within ± 15% of the theoretical concentration. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate. The acceptance criteria must be met on both columns.

10.6 Calibration Verification (CCV)

A CCV containing AR1660, at or below mid-calibration range, is analyzed each day before sample analysis, after every ten injections and at the end of each analytical batch to monitor instrument drift. The concentration of the CCV is varied. Calculate the CF and percent difference or drift (Appendix B) for each analyte on both columns. The percent difference or drift must be within ±15% for each analyte. Compare the RT of each analyte in the CCV with the RT windows; the RT must be within the window established in 10.4. The acceptance criteria must be met on both columns.

If the CCV fails, it may be repeated once. If it still fails, corrective action must be taken. The sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples must be bracketed by passing CCVs, and samples before and after CCV failure must be reanalyzed, unless the CCV is high and there are no detects in the associated samples.

10.7 Troubleshooting: the following items can be checked in case of calibration failures:

- ICAL Failure – Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if chromatography has degraded as evidenced by peak shapes.
- CCV Failure – Perform Injection port maintenance; if injection port maintenance does not restore CCV, install a new guard column.
- Needle crushed during injection - Replace the needle.
- Auto-sampler failure - Reset the auto-sampler.
- Power failure - Reset run in Multichrom and re-acquire or re-initiate run sequence.

11.0 PROCEDURE

11.1 Transfer approximately 100-µl of each QC standard and sample extract to an autosampler vial and place the vials in the auto-sampler. Arrange the samples in a sequence that begins with the calibration standards followed by the analysis of QC samples, field samples and continuing calibration verification standards (CCVs)

11.2 Arrange the samples in a sequence that begins with the calibration standards (if necessary) followed by the analysis of QC samples, field samples and continuing calibration verification standards (CCVs).

An example analysis sequence is given below:

Injection Number	Lab Description
1	Instrument Blank
2	AR1221 200PPB
3	AR1232 200PPB
4	AR1242 200PPB
5	AR1248 200PPB

6	AR1254 200PPB
7	AR1660 50PPB
8	AR1660 100PPB
9	AR1660 200PPB
10	AR1660 400PPB
11	AR1660 800PPB
12	ICV
13-22	10 injections
23	CCV
	Repeat until ending with CCV

11.3 Enter the sample ID's into the data acquisition program in the order the samples were placed in the autosampler and start the analytical sequence.

11.4 Cleaning blanks (CBLK) consisting of hexane may be analyzed after high-level samples at the discretion of the analyst.

11.5 The data system identifies the target analytes by comparing the retention time of the peaks to the retention times of the initial calibration standards. The data system does not recognize aroclor patterns. The analyst manually identifies aroclors by comparing the pattern in the samples to the patterns in the initial calibration standards. When "weathered" aroclor patterns are present, the laboratory identifies aroclors based on the best overall pattern match. Using an average of the chosen quantificaton peaks per aroclor, the data system calculates the corrected concentration for each target analyte from the calibration curve using the equations given in Appendix B. If sample interference is suspected, the laboratory may remove up to two quantificaton peaks per column. If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP LP-LB-0006 *Manual Integration.*

11.6 After analysis is complete, evaluate the results against the performance criteria given in Section 10 and Table 3, Section 18 and perform corrective action as necessary.

11.7 Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should ideally result in a determination within the upper half of the calibration curve.

12.0 CALCULATIONS

See Appendix B.

13.0 DATA ASSESSMENT, CORRECTIVE ACTION & REPORTING

13.1 Data Review and Corrective Action

Review the samples, standards and QC samples against the acceptance criteria in Table 3. If the results do not fall within the established limits, perform the recommended corrective action. If corrective action is unsuccessful, document the situation with a nonconformance report and/or qualify the data using an appropriate data qualifier (see Appendix C for data qualifier definitions). For additional guidance regarding the laboratory's protocol and required elements for each level of data review refer to laboratory SOP LP-LB-003 *Data Review*.

In the absence of project specific requirements, use the control limits specified in Table 1. The control limits in Table 1 are based on in-house statistically generated limits. In some cases, the in-house limits were outside of Department of Defense (DoD) limits as specified in the Quality Systems Manual for Environmental Laboratories. Where this is the case, the laboratory uses the stricter, DoD limits that are presented in bold in Table 1. For DoD projects, the in-house laboratory limits are also included in the project report.

Weathering of PCB's in the environment may alter the PCB's to the point that the pattern no longer matches the pattern established for that Aroclor in the initial calibration. The laboratory takes the best pattern match approach to the identification and quantification of weathered PCB's. In many cases, this entails choosing peaks such that the weathering pattern does not affect the quantificaton of the Aroclor.

13.2 Data Reporting

Unless otherwise specified, the higher result between the two columns is reported. The Relative Percent Difference (RPD) of the two results is calculated, and if the RPD is greater than 40% it is reported with a data flag. If, in the analyst's judgment, the higher result is due to overlapping peaks, or interference peaks, the lower of the two results should be reported with a data flag, and the issue discussed in the project narrative.

The laboratory's RL for each target analyte is provided in Table 1. Report the data to the RL adjusted for sample matrix, percent moisture, and sample dilution/concentration. The reporting limit is the threshold value below which results are reported as non-detected. Report sample results that have concentrations for a target analytes less than the RL with a "U" qualifier. Unless otherwise specified, report the results for solid matrices on a dry weight basis.

Some projects may require reporting positively identified target analytes less than the RL. In this case, the analyte can be qualitatively detected but not accurately quantified. Flag all results less than the RL with a "J" data qualifier (Appendix C).

Some projects may require RLs that are less than the laboratory's routine RL. Sample results may be reported to the project RL if the project RL is greater than the Quantification Limit (QL) and above the MDL. In this context, the QL is defined as the

concentration of the low calibration standard. If the project RL is less than the QL, all values less than the QL must be reported as estimated and qualified with a "J".

Further guidance on the application and use of the MDL, RL, and QL is provided in laboratory SOP LP-LB-009 *Determination of Method Detection Limits.*

13.3 Data Management and Records: All electronic and hardcopy data is managed, retained, and archived as specified in laboratory SOP LP-QA-0014 *Laboratory Records*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009 *Method Detection Limits.*

14.2 A demonstration of analyst capability (IDOC) is required before use of this SOP and any time there is a significant change in instrument type, personnel or test method.

14.3 Employee Training, and IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

14.4 The laboratory statistically derived control limits used to evaluate accuracy, precision and surrogate recoveries are provided in Table 1. The control limits for accuracy are based on compiled data and are set at 3 standard deviations around the mean using the procedures described in laboratory SOP LP-QA-012 *Control Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is carried out.

- Waste Solvents
- Solid Waste

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

Polychlorinated Biphenyls by Gas Chromatography (Method 8082), Revision 0; December 1996. USEPA SW-846 Methods for Evaluating Solid Waste, Update III.

17.0 SOP REVISION HISTORY

The following changes were made in this revision:

Section 6:	Added computer hardware and software and hydrogen generator.
Section 7:	7.1 - Removed solvents not used in analytical method.
Section 10:	10.4 - Changed RT window requirements to assess RT window from initial calibration curve. 10.6 Added detail about repeating CCV. 10.7 Added Troubleshooting.
Section 13:	13.1 - Added detail regarding the use of DoD LCS and Surrogate Limits. 13.2 – Moved dual column reporting from Section 11. Changed to reporting higher value and flagging at 40%. 13.3 - Added SOP reference for Data Management & Records.
Section 17:	New Section added.
Table 1:	Updated control limits. Removed TCMX as surrogate. Added footnotes.
Table 2:	Removed solvents not used in analytical method.
Table 3:	Re-worded corrective action for MB, MS/MSD, SD. Added detail to corrective action section.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

Table 1:	Target Analyte List, Reporting Limits, and Control Limits as Accuracy (%R) and Precision (RPD)
Table 2:	Primary Materials Used
Table 3:	QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action
Appendix A:	Standard Preparation Tables
Appendix B:	Equations
Appendix C:	Terms & Definitions

Table 1: Target Analyte List, Reporting Limits[1], and Control Limits[2] as Accuracy (%R) and Precision[4] (RPD)

Analyte	Reporting Limit[1]		Water		Solid, Chemical Material, Tissue	
	Water ug/L	Soil ug/Kg	%R	RPD[4]	%R	RPD[3]
AR1016	0.50	17	55-125	≤ 35	55-120	≤ 30
AR1221	0.50	17	NA	NA	NA	NA
AR1232	0.50	17	NA	NA	NA	NA
AR1242	0.50	17	NA	NA	NA	NA
AR1248	0.50	17	NA	NA	NA	NA
AR1254	0.50	17	NA	NA	NA	NA
AR1260	0.50	17	55-120	≤ 30	60-130	≤ 35
DCB (Surrogate)	NA	NA	55-120	NA	60-125	NA

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.
[2] The in-house statistical control limits posted in this table are those in effect on the revision date of this SOP. These limits are subject to change based on performance trends.
[3] RPD for MS/MSD only.

Table 2: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

[1] Always add acid to water to prevent violent reactions.
[2] Exposure limit refers to the OSHA regulatory exposure limit.

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CF: RSD ≤ 20% Linear Regression: r ≥ 0.99 (0.995 for DoD)	Correct problem, reanalyze, repeat calibration.
ICV	After each initial calibration	%Difference ± 15% from expected value	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±15%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and bracketed samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	< RL DoD: ≤ ½ RL If analyte in any sample ≥ RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	Evaluated against control limits in Table 1	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch SD: Per client request	Evaluated against control limits in Table 1	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate Spike	All field and QC samples	Evaluated against control limits in Table 1	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

Appendix A: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory SOP LP-LB-002 *Standard Preparation* for further guidance on the preparation of standard solutions.

All standards are prepared using volumetric glassware, including Class A volumetric flasks, and Hamilton Syringes. Unless otherwise noted, or unless the expiration date of the parent standard is earlier, an expiration date of 6 months from date of preparation is assigned to all intermediate and working standards. If the expiration date of any of parent standards is earlier, that earlier expiration date is used.

Table Legend:
C_{stock} = Concentration of Parent Standard
V_{stock} = Volume of Parent Standard
V_{std} = Volume of Prepared Standard
C_{std} = Theoretical Concentration of Prepared Standard

INTERMEDIATE STANDARDS – in hexane

Intermediate Standards-10mg/L each: AR1660, AR1254, AR1248, AR1242, AR1232, AR1221 These are prepared as 6 individual standards in hexane.

Stock Standard	Restek Catalog #	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (mg/L)
AR1016/AR1260[1]	32039	1000	0.400	40	10
AR1254	32011	1000	0.400	40	10
AR1248	32010	1000	0.400	40	10
AR1242	32009	1000	0.400	40	10
AR1232	32008	1000	0.400	40	10
AR1221	32007	1000	0.400	40	10

[1] Concentration represents concentration of each Aroclor in mixed standard rather than concentration of both.

ICV – Second Source Standard - AR1660 10mg/L

Stock Standard	Restek Catalog #	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (mg/L)
AR1016/AR1260	32039*	1000	0.400	40	10

* ***Must*** be from a different lot than calibration standards!

WORKING STANDARDS – in hexane

DCB 10mg/L

Stock Standard	Restek Catalog #	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (mg/L)
Pesticide Surrogate	32000	1000	0.400	40	10

CALIBRATION STANDARDS – in hexane

AR1660 Calibration Level 5 - 800 μg/L

Stock Standard	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (μg/L)
AR1660 Intermediate Standard	10	8.0	100	800
DCB Working Standard	10	0.800	100	80

Prepare Calibration Standards Level 1-4 in hexane using AR1660 Calibration Level 5 as stock:

AR1660 Calibration Working Standards (Level 1-4)

AR1660 800 ppb (Calibration Level 5)	400 ppb	200 ppb	100 ppb	50 ppb
V_{stock} mL	20	25	5.0	2.5
V_{std} mL	40	100	40	40

AR1254 Working 200μg/L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1254 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1248 Working 200μg /L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1248 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1242 Working 200μg /L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1242 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1232 Working 200μg /L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1232 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1221 Working 200ug/L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1221 Intermediate	10	800	40	200
DCB Working	10	80	40	20

ICV – Second Source Standard AR1660 Working 200ug/L

Stock Standard	C_{stock} (mg/L)	V_{stock} μL	V_{std} mL	C_{std} (μg/L)
AR1660 Intermediate	10	1000	50	200
DCB Working	10	100	50	20

Final Concentration of Prepared Calibration Standards

Analyte	Level 1 ug/L	Level 2 ug/L	Level 3 ug/L	Level 4 ug/L	Level 5 ug/L
DCB (Surrogate)	5	10	20	40	80
AR1660	50	100	200	400	800
AR1254	NA	NA	200	NA	NA
AR1248	NA	NA	200	NA	NA
AR1242	NA	NA	200	NA	NA
AR1232	NA	NA	200	NA	NA
AR1221	NA	NA	200	NA	NA

Appendix B: Equations

Calibration Factor (CF_x) = $\frac{\text{Peak area or height}_{(x)}}{\text{Standard concentration}_{(ug/L)}}$

Mean Calibration Factor ($\overline{CF}$) = $\frac{\sum_{i=1}^{n} CF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Calibration Factor (SD) = $\sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Calibration Factor = $\frac{SD}{\overline{CF}} \times 100\%$

Percent Difference (%D) = $\frac{CF_v - \overline{CF}}{\overline{CF}} \times 100\%$

where: CF_v = Calibration Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $\frac{\text{Calculated Concentration – Theoretical Concentration}}{\text{Theoretical Concentration}}$ X 100%

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD) = $\frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$$C_{extract}\,(ug/L) = \frac{\text{Peak Area (or Height)}}{\overline{CF}}$$

Note: The concentrations of the 3-5 peaks chosen for quantificaton is calculated and the average is then taken for final calculation.

Water

$$C_{sample}\,(ug/L) = C_{extract}\,(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample volume (L)}} \times DF$$

Solids

$$C_{sample}\,(ug/Kg) = C_{extract}\,(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample weight (Kg)}} \times \frac{100}{\%\ \text{solids}} \times DF$$

where: DF = Extract Dilution Factor. If no dilution was made, DF=1.

Appendix C: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

The qualifiers that are routinely used for this test method are:

U: Compound analyzed for but not detected at a concentration above the reporting limit.
J: Estimated Value
P: There is greater 40% difference for detected concentrations between two GC columns
C: Positive result whose identification has been confirmed by GC/MS
B: Compound is found in the sample and the associated method blank.
E: Compound whose concentration exceeds the upper limit of the calibration range.
D: Concentration identified from a dilution analysis.

X,Y,Z: Laboratory defined flags that may be used alone or combined as needed. If used, provide a description of the flag in the project narrative.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

STANDARD OPERATING PROCEDURE
POLYCHLORINATED BIPHENYLS (PCB'S) BY GC/ECD
SW-846 METHOD 8082
Applicable Matrices: Non-Potable Water, Solid and Chemical Materials
Standard Compound List and Reporting Limits: See Table 1

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the determination of concentrations of Polychlorinated Biphenyls (PCBs) in extracts derived from non-potable water, solids, tissue, air, and chemical materials including TCLP leachates, using dual column Gas Chromatography with Electron Capture Detectors (GC/ECD). This SOP is applicable to the analytical procedure only; the extraction and extract cleanup methods referenced in this SOP are described in the following laboratory SOPs:

LM-OP-3510	Separatory Funnel Extraction
LM-OP-3540	Soxhlet Extraction
LM-OP-3541	Automated Soxhlet Extraction
LM-OP-3550	Ultrasonic Extraction
LM-OP-Cleanup	Extract Cleanup Procedures
LM-OP-GPC	Gel Permeation Chromatography (GPC)

1.2 The analytes that can be determined by this procedure and their associated Reporting Limits (RL) are listed in Table 1, Section 18.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using an appropriate matrix-specific extraction technique. After extraction, the extract may be subject to cleanup depending on the nature of sample matrix and the target analytes. After cleanup, the extract is analyzed by injecting a 2-µl aliquot into a dual capillary column GC/ECD.

2.2 This procedure is based on SW-846 Method 8082, Revision 0, December 1996.

3.0 DEFINITIONS

A list of terms and definitions is given in Appendix C.

4.0 INTERFERENCES

4.1 Contaminated solvents, reagents or equipment can cause interferences. To reduce the occurrence of this type of interference, glassware must be cleaned thoroughly before use following the procedure given in laboratory SOP LM-OP-Glass *Glassware Washing*. Solvents and acids are lot- tested and approved for use before delivery to the laboratory in accordance with STL-T-001 *Testing of Solvent and Acids*. Solvents should be stored in an area away from organochlorine compounds to minimize contamination.

4.2 Phthalate esters introduced during sample preparation can pose a problem in the determination of pesticides. Common flexible plastics contain varying amounts of phthalate esters, and these can be easily extracted or leached during extraction. To minimize this interference, avoid contact with any plastic materials.

4.3 Non-target compounds co-extracted from the sample matrix can also cause interference, the extent of which will vary considerably depending on the nature of the samples. Elemental sulfur is often found in sediment samples and its presence will result in broad peaks. Samples are screened before analysis and those samples that contain high levels of sulfur are subject to cleanup using activated copper before analysis (SW-846 3660B). Waxes, lipids, other high molecular weight materials and co-eluting organophosphorous pesticides may be removed by extract cleanup with GPC (SW-846-3640A). Co-eluting chlorophenols can be eliminated by cleanup with silica gel (SW-846 3630C), or Florisil (SW-846 3620B), or Sulfuric acid Cleanup (SW-846 3665A) may be used to eliminate certain organochlorine pesticides and elevated baselines.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 Specific Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 Primary Materials Used

Table 2, Section 18 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. Note: The table does not include all materials used in the procedure. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used can be found in Section 7. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 EQUIPMENT AND SUPPLIES

6.1 Autosampler Vials, National Scientific or equivalent.

6.2 Computer Hardware/Software: GC Acquisition Platform - VAX 4505 (GVAX) Multichrom V2.11. Data Processing - Hewlett-Packard 9000-series computers, an HP9000 D250 (Chemsvr4) and an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5.

6.3 GC/ECD: with dual columns, dual ECDs, and auto-sampler capable of a 2-µl injection split onto two columns: HP 5890 with Leap Technology CTC A200SE and A200S Fisons autosamplers, or equivalent.

6.4 GC Columns: A dual fused silica capillary column system that will provide simultaneous primary and confirmation analyses.

- RTX-5, (30m x 0.25 mmID x 0.25um)
- RTX-35, (30m x 0.25 mmID x 0.25um)
- Equivalent columns may be used, provided the elution orders are documented and compound separations are maintained.

6.5 Hydrogen Generator: Whatman.

6.6 Volumetric Syringes, Class "A" (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

- Hexane JT Baker Ultra-Resi analyzed.

7.2 Standards

Stock standard solutions are purchased made from commercial vendors. Intermediate and working standards solutions are prepared in the laboratory by diluting a known volume of stock standard in an appropriate solvent and diluting to a specified volume. Standard preparation procedures for intermediate and working standard solutions are provided in Appendix A.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 Sample extracts must be stored at 4°C ± 2° until the time of analysis. The analytical holding time is 40 days from date of sample extraction.

8.2 Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 The minimum frequency requirements, acceptance criteria and recommended corrective action for all QC samples are summarized in Section 18, Table 3. Below is a summary of each type of QC sample that is analyzed with the method.

9.2 A Method Blank (MB) and Laboratory Control Sample (LCS) are prepared with each extraction batch. These samples show that the laboratory is in control, independent of the sample matrix.

9.3 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) are prepared with each extraction batch. Project specific MS/MSD are performed per client request. Sample Duplicates (SD) are performed per client request. These samples show the effect of the sample matrix on the accuracy and precision of the method.

9.4 A Surrogate spike is added to all field and QC samples before extraction to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix.

9.5 Instrumental QC standards include a five-point ICAL is generated for Aroclor 1016 and 1260 (referred to as AR1660) and an initial one point calibration for all other Aroclors. After the ICAL, an Initial Calibration Verification (ICV) standard, also referred to as a second source standard, containing Aroclor 1660 is analyzed to verify the ICAL standard formulation. Continuing Calibration Verification (CCV) standards are analyzed before sample analysis, every ten samples thereafter, and at the end of the run to assess instrument drift.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Operating Conditions

Install a five meter deactivated guard column to the injection port and connect the guard column to the separate analytical columns using a glass “Y”. Then attach the analytical columns to the dual ECD detectors.

The recommended instrument operating conditions are as follows:

Initial Temperature:	130°C for 1 minute
Temperature Program:	20°C per minute to 190°C to 5°C per minute to 225°C to 20.0°C per minute to 300°C. Hold for 6 minutes.
Detector Temperature	310°C
Injector Temperature:	225°C
Injection volume:	2μL
Carrier Gas:	Hydrogen (supplied by hydrogen generators)

Optimize the flow rate of the carrier gas by injecting an un-retained substance onto the column at an isothermal oven state and adjusting the flow to obtain the recommended dead volume time.

10.2 Initial Calibration

Before initial or daily calibration, inject an instrument blank (IBLK) consisting of hexane blank to bring the GC/ECD system online.

A multi-point calibration of AR1660 at five concentrations is sufficient in demonstrating linearity. AR1660 includes most of the peaks represented in the other five Aroclors. For the remaining Aroclors, a midlevel standard is analyzed to aid in pattern recognition and is used as a single point calibration standard. Five point calibrations for these aroclors

will only be performed when required by the client or when specified as a regulatory requirement. A minimum of three to five peaks per aroclor is used for quantificaton. The calibration standards are introduced using the same technique that is used for sample extracts described in Section 11.

Inject 2-µl of each calibration standard and calculate the Calibration Factor (CF), mean CF and Percent Relative Standard Deviation (% RSD) for each analyte on both columns (Appendix B). The %RSD for each peak must be ≤ 20 % for the curve to be considered acceptable. If any of the results are outside QC criteria, the cause of the problem is investigated and corrected prior to analysis of samples.

10.3 Alternate Quantificaton. In some cases, it may be preferable to use linear regression to quantify the compounds. The following approaches may be used:

Linear Regression - A curve of concentration vs. peak area is generated for each analyte and the correlation coefficient is calculated. The calibration must have a correlation coefficient (r) ≥ 0.99 (0.995 for DoD) for acquisition of samples to continue. The use of linear regression requires a minimum of 5 calibration points. See SW-846 Method 8000B for linear regression calculations.

Once a method of calibration is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is generated.

10.4 Retention Time Windows

When a new GC column is installed, RT windows are established for 3-5 peaks for Aroclor 1660 by analyzing three standards over a 72-hour period and calculating the mean RT and Standard Deviation (SD). The RT window is calculated as mean RT ± 3SD of the three standards. If the SD is <0.01 minutes, the laboratory may use a default SD of 0.01 minutes.

If, in the professional judgment of the analyst, this results in an RT window that is too tight and would favor false negatives, the laboratory may opt use an alternate method to determine the RT windows as follows: using the RT of the midpoint initial calibration standard, calculate the RT window using ± 0.05 minutes from the midpoint of the RT in the initial calibration.

10.5 ICV – Second Source Standard

After each calibration, verify the accuracy of the initial calibration by analyzing the ICV (Appendix A). The calculated concentration of each analyte must be within ± 15% of the theoretical concentration. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate. The acceptance criteria must be met on both columns.

10.6 Calibration Verification (CCV)

A CCV containing AR1660, at or below mid-calibration range, is analyzed each day before sample analysis, after every ten injections and at the end of each analytical batch to monitor instrument drift. The concentration of the CCV is varied. Calculate the CF and percent difference or drift (Appendix B) for each analyte on both columns. The percent difference or drift must be within ±15% for each analyte. Compare the RT of each analyte in the CCV with the RT windows; the RT must be within the window established in 10.4. The acceptance criteria must be met on both columns.

If the CCV fails, it may be repeated once. If it still fails, corrective action must be taken. The sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples must be bracketed by passing CCVs, and samples before and after CCV failure must be reanalyzed, unless the CCV is high and there are no detects in the associated samples.

10.7 Troubleshooting: the following items can be checked in case of calibration failures:

- ICAL Failure – Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if chromatography has degraded as evidenced by peak shapes.
- CCV Failure – Perform Injection port maintenance; if injection port maintenance does not restore CCV, install a new guard column.
- Needle crushed during injection - Replace the needle.
- Auto-sampler failure - Reset the auto-sampler.
- Power failure - Reset run in Multichrom and re-acquire or re-initiate run sequence.

11.0 PROCEDURE

11.1 Transfer approximately 100-µl of each QC standard and sample extract to an autosampler vial and place the vials in the auto-sampler. Arrange the samples in a sequence that begins with the calibration standards followed by the analysis of QC samples, field samples and continuing calibration verification standards (CCVs)

11.2 Arrange the samples in a sequence that begins with the calibration standards (if necessary) followed by the analysis of QC samples, field samples and continuing calibration verification standards (CCVs).

An example analysis sequence is given below:

Injection Number	Lab Description
1	Instrument Blank
2	AR1221 200PPB
3	AR1232 200PPB
4	AR1242 200PPB
5	AR1248 200PPB

6	AR1254 200PPB
7	AR1660 50PPB
8	AR1660 100PPB
9	AR1660 200PPB
10	AR1660 400PPB
11	AR1660 800PPB
12	ICV
13-22	10 injections
23	CCV
	Repeat until ending with CCV

11.3 Enter the sample ID's into the data acquisition program in the order the samples were placed in the autosampler and start the analytical sequence.

11.4 Cleaning blanks (CBLK) consisting of hexane may be analyzed after high-level samples at the discretion of the analyst.

11.5 The data system identifies the target analytes by comparing the retention time of the peaks to the retention times of the initial calibration standards. The data system does not recognize aroclor patterns. The analyst manually identifies aroclors by comparing the pattern in the samples to the patterns in the initial calibration standards. When "weathered" aroclor patterns are present, the laboratory identifies aroclors based on the best overall pattern match. Using an average of the chosen quantificaton peaks per aroclor, the data system calculates the corrected concentration for each target analyte from the calibration curve using the equations given in Appendix B. If sample interference is suspected, the laboratory may remove up to two quantificaton peaks per column. If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP LP-LB-0006 *Manual Integration.*

11.6 After analysis is complete, evaluate the results against the performance criteria given in Section 10 and Table 3, Section 18 and perform corrective action as necessary.

11.7 Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should ideally result in a determination within the upper half of the calibration curve.

12.0 CALCULATIONS

See Appendix B.

13.0 DATA ASSESSMENT, CORRECTIVE ACTION & REPORTING

13.1 Data Review and Corrective Action

Review the samples, standards and QC samples against the acceptance criteria in Table 3. If the results do not fall within the established limits, perform the recommended corrective action. If corrective action is unsuccessful, document the situation with a nonconformance report and/or qualify the data using an appropriate data qualifier (see Appendix C for data qualifier definitions). For additional guidance regarding the laboratory's protocol and required elements for each level of data review refer to laboratory SOP LP-LB-003 *Data Review*.

In the absence of project specific requirements, use the control limits specified in Table 1. The control limits in Table 1 are based on in-house statistically generated limits. In some cases, the in-house limits were outside of Department of Defense (DoD) limits as specified in the Quality Systems Manual for Environmental Laboratories. Where this is the case, the laboratory uses the stricter, DoD limits that are presented in bold in Table 1. For DoD projects, the in-house laboratory limits are also included in the project report.

Weathering of PCB's in the environment may alter the PCB's to the point that the pattern no longer matches the pattern established for that Aroclor in the initial calibration. The laboratory takes the best pattern match approach to the identification and quantification of weathered PCB's. In many cases, this entails choosing peaks such that the weathering pattern does not affect the quantificaton of the Aroclor.

13.2 Data Reporting

Unless otherwise specified, the higher result between the two columns is reported. The Relative Percent Difference (RPD) of the two results is calculated, and if the RPD is greater than 40% it is reported with a data flag. If, in the analyst's judgment, the higher result is due to overlapping peaks, or interference peaks, the lower of the two results should be reported with a data flag, and the issue discussed in the project narrative.

The laboratory's RL for each target analyte is provided in Table 1. Report the data to the RL adjusted for sample matrix, percent moisture, and sample dilution/concentration. The reporting limit is the threshold value below which results are reported as non-detected. Report sample results that have concentrations for a target analytes less than the RL with a "U" qualifier. Unless otherwise specified, report the results for solid matrices on a dry weight basis.

Some projects may require reporting positively identified target analytes less than the RL. In this case, the analyte can be qualitatively detected but not accurately quantified. Flag all results less than the RL with a "J" data qualifier (Appendix C).

Some projects may require RLs that are less than the laboratory's routine RL. Sample results may be reported to the project RL if the project RL is greater than the Quantification Limit (QL) and above the MDL. In this context, the QL is defined as the

concentration of the low calibration standard. If the project RL is less than the QL, all values less than the QL must be reported as estimated and qualified with a "J".

Further guidance on the application and use of the MDL, RL, and QL is provided in laboratory SOP LP-LB-009 *Determination of Method Detection Limits.*

13.3 Data Management and Records: All electronic and hardcopy data is managed, retained, and archived as specified in laboratory SOP LP-QA-0014 *Laboratory Records*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009 *Method Detection Limits.*

14.2 A demonstration of analyst capability (IDOC) is required before use of this SOP and any time there is a significant change in instrument type, personnel or test method.

14.3 Employee Training, and IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

14.4 The laboratory statistically derived control limits used to evaluate accuracy, precision and surrogate recoveries are provided in Table 1. The control limits for accuracy are based on compiled data and are set at 3 standard deviations around the mean using the procedures described in laboratory SOP LP-QA-012 *Control Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is carried out.

- Waste Solvents
- Solid Waste

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

Polychlorinated Biphenyls by Gas Chromatography (Method 8082), Revision 0; December 1996. USEPA SW-846 Methods for Evaluating Solid Waste, Update III.

17.0 SOP REVISION HISTORY

The following changes were made in this revision:

Section 6:	Added computer hardware and software and hydrogen generator.
Section 7:	7.1 - Removed solvents not used in analytical method.
Section 10:	10.4 - Changed RT window requirements to assess RT window from initial calibration curve. 10.6 Added detail about repeating CCV. 10.7 Added Troubleshooting.
Section 13:	13.1 - Added detail regarding the use of DoD LCS and Surrogate Limits. 13.2 – Moved dual column reporting from Section 11. Changed to reporting higher value and flagging at 40%. 13.3 - Added SOP reference for Data Management & Records.
Section 17:	New Section added.
Table 1:	Updated control limits. Removed TCMX as surrogate. Added footnotes.
Table 2:	Removed solvents not used in analytical method.
Table 3:	Re-worded corrective action for MB, MS/MSD, SD. Added detail to corrective action section.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

Table 1:	Target Analyte List, Reporting Limits, and Control Limits as Accuracy (%R) and Precision (RPD)
Table 2:	Primary Materials Used
Table 3:	QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action
Appendix A:	Standard Preparation Tables
Appendix B:	Equations
Appendix C:	Terms & Definitions

Table 1: Target Analyte List, Reporting Limits[1], and Control Limits[2] as Accuracy (%R) and Precision[4] (RPD)

Analyte	Reporting Limit[1]		Water		Solid, Chemical Material, Tissue	
	Water ug/L	Soil ug/Kg	%R	RPD[4]	%R	RPD[3]
AR1016	0.50	17	55-125	≤ 35	55-120	≤ 30
AR1221	0.50	17	NA	NA	NA	NA
AR1232	0.50	17	NA	NA	NA	NA
AR1242	0.50	17	NA	NA	NA	NA
AR1248	0.50	17	NA	NA	NA	NA
AR1254	0.50	17	NA	NA	NA	NA
AR1260	0.50	17	55-120	≤ 30	60-130	≤ 35
DCB (Surrogate)	NA	NA	55-120	NA	60-125	NA

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.
[2] The in-house statistical control limits posted in this table are those in effect on the revision date of this SOP. These limits are subject to change based on performance trends.
[3] RPD for MS/MSD only.

Table 2: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

[1] Always add acid to water to prevent violent reactions.
[2] Exposure limit refers to the OSHA regulatory exposure limit.

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Correct
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CF: RSD ≤ 20% Linear Regression: r ≥ 0.99 (0.995 for DoD)	Correct problem, reanalyze, repeat ca
ICV	After each initial calibration	%Difference ± 15% from expected value	Correct problem and verify second so fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±15%	Re-analyze once, if still outside criteri action, sequence can be re-started if t pass, otherwise repeat ICAL and all a since last successful CCV, unless CC bracketed samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	< RL DoD: ≤ ½ RL If analyte in any sample ≥ RL	Examine project DQO's and take app action, which may include re-analysis batch, and/or non-conformance repor action must be documented on NCR. in samples, or if all detects are > 10 X reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	Evaluated against control limits in Table 1	Examine project DQO's and take app action, which may include re-analysis of batch, and/or non-conformance rep action must be documented on NCR. values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch SD: Per client request	Evaluated against control limits in Table 1	Evaluate data and determine if a matr error is indicated. If analytical error, re extract. Flag all reported values outsi
Surrogate Spike	All field and QC samples	Evaluated against control limits in Table 1	Evaluate data and determine if a matr error is indicated. If analytical error, re If matrix effect, review project DQOs t effect must be confirmed by re-analys values outside of control limits.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

Appendix A: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory SOP LP-LB-002 *Standard Preparation* for further guidance on the preparation of standard solutions.

All standards are prepared using volumetric glassware, including Class A volumetric flasks, and Hamilton Syringes. Unless otherwise noted, or unless the expiration date of the parent standard is earlier, an expiration date of 6 months from date of preparation is assigned to all intermediate and working standards. If the expiration date of any of parent standards is earlier, that earlier expiration date is used.

Table Legend:
C_{stock} = Concentration of Parent Standard
V_{stock} = Volume of Parent Standard
V_{std} = Volume of Prepared Standard
C_{std} = Theoretical Concentration of Prepared Standard

INTERMEDIATE STANDARDS – in hexane

Intermediate Standards-10mg/L each: AR1660, AR1254, AR1248, AR1242, AR1232, AR1221 These are prepared as 6 individual standards in hexane.

Stock Standard	Restek Catalog #	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (mg/L)
AR1016/AR1260[1]	32039	1000	0.400	40	10
AR1254	32011	1000	0.400	40	10
AR1248	32010	1000	0.400	40	10
AR1242	32009	1000	0.400	40	10
AR1232	32008	1000	0.400	40	10
AR1221	32007	1000	0.400	40	10

[1] Concentration represents concentration of each Aroclor in mixed standard rather than concentration of both.

ICV – Second Source Standard - AR1660 10mg/L

Stock Standard	Restek Catalog #	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (mg/L)
AR1016/AR1260	32039*	1000	0.400	40	10

* ***Must*** be from a different lot than calibration standards!

WORKING STANDARDS – in hexane

DCB 10mg/L

Stock Standard	Restek Catalog #	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (mg/L)
Pesticide Surrogate	32000	1000	0.400	40	10

CALIBRATION STANDARDS – in hexane

AR1660 Calibration Level 5 - 800 µg/L

Stock Standard	C_{stock} (mg/L)	V_{stock} mL	V_{std} mL	C_{std} (µg/L)
AR1660 Intermediate Standard	10	8.0	100	800
DCB Working Standard	10	0.800	100	80

Prepare Calibration Standards Level 1-4 in hexane using AR1660 Calibration Level 5 as stock:

AR1660 Calibration Working Standards (Level 1-4)

AR1660 800 ppb (Calibration Level 5)	400 ppb	200 ppb	100 ppb	50 ppb
V_{stock} mL	20	25	5.0	2.5
V_{std} mL	40	100	40	40

AR1254 Working 200µg/L

Stock Standard	C_{stock} (mg/L)	V_{stock} µL	V_{std} mL	C_{std} (µg/L)
AR1254 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1248 Working 200µg /L

Stock Standard	C_{stock} (mg/L)	V_{stock} µL	V_{std} mL	C_{std} (µg/L)
AR1248 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1242 Working 200µg /L

Stock Standard	C_{stock} (mg/L)	V_{stock} µL	V_{std} mL	C_{std} (µg/L)
AR1242 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1232 Working 200µg /L

Stock Standard	C_{stock} (mg/L)	V_{stock} µL	V_{std} mL	C_{std} (µg/L)
AR1232 Intermediate	10	800	40	200
DCB Working	10	80	40	20

AR1221 Working 200ug/L

Stock Standard	C_{stock} (mg/L)	V_{stock} µL	V_{std} mL	C_{std} (µg/L)
AR1221 Intermediate	10	800	40	200
DCB Working	10	80	40	20

ICV – Second Source Standard AR1660 Working 200ug/L

Stock Standard	C_{stock} (mg/L)	V_{stock} µL	V_{std} mL	C_{std} (µg/L)
AR1660 Intermediate	10	1000	50	200
DCB Working	10	100	50	20

Final Concentration of Prepared Calibration Standards

Analyte	Level 1 ug/L	Level 2 ug/L	Level 3 ug/L	Level 4 ug/L	Level 5 ug/L
DCB (Surrogate)	5	10	20	40	80
AR1660	50	100	200	400	800
AR1254	NA	NA	200	NA	NA
AR1248	NA	NA	200	NA	NA
AR1242	NA	NA	200	NA	NA
AR1232	NA	NA	200	NA	NA
AR1221	NA	NA	200	NA	NA

Appendix B: Equations

Calibration Factor (CF_x) = $\frac{\text{Peak area or height}_{(x)}}{\text{Standard concentration}_{(ug/L)}}$

Mean Calibration Factor ($\overline{CF}$) = $\frac{\sum_{i=1}^{n} CF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Calibration Factor (SD) = $\sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Calibration Factor = $\frac{SD}{\overline{CF}} \times 100\%$

Percent Difference (%D) = $\frac{CF_v - \overline{CF}}{\overline{CF}} \times 100\%$

where: CF_v = Calibration Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $\frac{\text{Calculated Concentration – Theoretical Concentration}}{\text{Theoretical Concentration}}$ X 100%

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD) = $\frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$$C_{extract}\,(ug/L) = \frac{\text{Peak Area (or Height)}}{\overline{CF}}$$

Note: The concentrations of the 3-5 peaks chosen for quantificaton is calculated and the average is then taken for final calculation.

Water

$$C_{sample}\,(ug/L) = C_{extract}\,(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample volume (L)}} \times DF$$

Solids

$$C_{sample}\,(ug/Kg) = C_{extract}\,(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample weight (Kg)}} \times \frac{100}{\%\ \text{solids}} \times DF$$

where: DF = Extract Dilution Factor. If no dilution was made, DF=1.

Appendix C: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

The qualifiers that are routinely used for this test method are:

U: Compound analyzed for but not detected at a concentration above the reporting limit.
J: Estimated Value
P: There is greater 40% difference for detected concentrations between two GC columns
C: Positive result whose identification has been confirmed by GC/MS
B: Compound is found in the sample and the associated method blank.
E: Compound whose concentration exceeds the upper limit of the calibration range.
D: Concentration identified from a dilution analysis.

X,Y,Z: Laboratory defined flags that may be used alone or combined as needed. If used, provide a description of the flag in the project narrative.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

A FRESH APPROACH TO AROCLOR ANALYSIS

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ABSTRACT

This paper outlines an analytical approach to optimize data handling in multicomponent group analyses. Although the analytical techniques outlined in the paper are not specific and can be applied to other multicomponent analytical problems, an implementation for PCBs is described.

Traditional PCB methods quantify Aroclor mixtures by comparing the GC/ECD response of samples to responses of appropriate standards. Depending on the origin and state of environmental alteration of PCBs in samples, picking a standard may be simple or complicated. PCB data used in remediation often require validation. The EPA CLP program and more recently SW846 have proposed methods where quantitation is based on averages of results using 3 to 5 major peaks from dual capillary column data. This approach to calculation is simple to validate but can provide wild quantitative results if samples are environmentally altered.

A mathematical modeling scheme for Aroclor calibration data is presented which incorporates all usable peaks on both primary and secondary capillary GC columns to arrive at optimum qualitative and quantitative results. The method is useful even for complicated mixtures of pesticides and PCBs. A mixture of 100 ppb Aroclor 1242, 1254 and 1260 was identified and quantified within 20 % of true value in the presence of 200 ppb technical chlordane, 1000 ppb toxaphene and 40-80 ppb of CLP pesticide mixtures INDA and INDB.

As the approach in this paper is a large computer application, the validation of the data needs to be approached differently. A straightforward validation scheme is presented which gives the new technique more widespread utility.

INTRODUCTION

Multicomponent analyte mixtures are common in environmental testing. Among the chlorinated hydrocarbons, multicomponent analytes include polychlorinated biphenyls (PCBs), toxaphene and technical chlordane. Among the non-halogenated hydrocarbons, we find gasoline, diesel fuel and kerosene. These mixtures can be identified and quantified by gas chromatography using dual capillary columns. The first column and second column are chosen to give different separations for the same mixture. The qualitative and quantitative information from the second column can be used to confirm the results of the first column. The high resolving power of each capillary column usually results in many separated peaks for mixtures with a large number of components.

These mixtures pose challenges for a laboratory and limit the potential for automation. Any multicomponent analyte is subject to qualitative and quantitative complications in analysis and the effort involved in proper manual identification and quantitation can be considerable. Often simplifying assumptions are made which needlessly compromise true data quality.

While the widespread use of capillary columns has increased the potential for accuracy in multicomponent analysis, adequate tools must be employed to take advantage of the increased information. Often these tools are either not used or not available. Quantitative schemes geared towards the older packed columns can lead to serious errors when misapplied to capillary work, especially if most of the capillary information is ignored in an effort to minimize complexity of properly dealing with large numbers of peaks. Quantitative calculations may be performed on a selected subset of all the peaks available. Depending on how peaks are selected, different analysts can come up with widely differing results for the same original full data set.

Interferences pose problems in multicomponent work. Basing quantification on automated schemes involving preselected peaks is not always practical. When an interference prevents use of one or more of the preselected peaks, alternatives include use of other peaks to manually recalculate results after data review or flagging the original results between the primary and secondary analytical columns as not being reproducible. Hand interaction and manual calculation schemes reduce sample throughput in the laboratory and increase the potential for calculation errors.

PCBS

PCBs are polychlorinated biphenyls. There are 209 unique compounds or congeners in this family, each containing one or more chlorine atoms in substitution for hydrogen on the biphenyl ring. They were widely used before the late 1970s in mixtures containing various weight percentages of chlorine. Monsanto was a US manufacturer of PCB mixtures and sold them under the trade name Aroclor. Aroclor 1221 was a mixture with an average chlorination of 21%, 1242 was 42% chlorine etc. An exception to the naming was Aroclor 1016 which was similar to Aroclor 1242 but had a reduced concentration of some of the higher molecular weight congeners. PCBs are commonly quantified by expressing unknown sample chromatograms in terms of one or more standard Aroclors.

ANALYTICAL APPROACH

EPA's SW846 method 8080A(1) states:

> *"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 judgment about what proportion of the different Aroclors to combine to produce the appropriate reference material."*

Since PCBs are reported in terms of standard Aroclor mixtures, selection of standards matching samples becomes critical to accurate quantitation. On a practical level this can be difficult because the composition of unknowns is typically not characterized at the time of calibration. To further complicate matters, samples can be environmentally altered mixtures of one or more Aroclors. These alterations are often selective and can effect one individual peak more than a group of others. An automated scheme has been designed incorporating three modules which systematically circumvent these problems. The system is designed to express unknown sample chromatograms as linear combinations of the standard Aroclors. The "most similar" criterion of method 8080A is met by a least squares minimization of the difference between the unknown chromatogram and linear combinations of the Aroclors.

In this discussion, *i* refers to peaks, *j* refers to Aroclors, *k* refers to columns/detectors and *l* refers to concentration levels. To illustrate the principle, a program is used to determine specific retention time windows for PCB peaks in each standard Aroclor mixture (j) for each column. Five calibration points are obtained for each peak and a response factor (RF_{ij}) is computed for each peak group (using equation 10) in units of peak response (h_i') over concentration. When an unknown sample is analyzed, the program looks for any peaks in the predetermined window and if any are found they become the set of peaks (i) to be used for quantitation. The goodness-of-fit, how well a set of Aroclors at concentration c_j account for an unknown set of peaks h_i' is given by summing the differences between h_i' and the added concentrations of the contributions of each of the set of Aroclors ($c_1 RF_{i,1} + c_2 RF_{i,2} + \ldots + c_n RF_{in}$) over all of the Aroclor peaks on both columns. The "best fit", then is that which minimizes this goodness-of-fit.

More formally, given an unknown set S of n_i peaks of height $h_{i,k}$, we wish to estimate concentrations $\vec{\hat{c}} = \left(\hat{c}_1, \hat{c}_2, \ldots, \hat{c}_{n_j}\right)$ of n_j mixtures (i.e. Aroclors) such that the error E_{id}:

$$E_{id} = \sum_k \sum_i \left[\left(\sum_j \hat{c}_j a_{i,j,k} \right) - h_{i,k}^{\lambda_k} \right]^2 \qquad (1)$$

is minimized, subject to the constraint $\hat{c}_j \geq 0$. This minimization of sums-of-squares of residuals is the operative definition of goodness-of-fit. Implementation of this model requires determination of the λ_k-- the linearization constant for each n_k detector-column pair and $a_{i,j,k}$ --the response factors for each peak of each detector and for each possible mixture. Equation (1) is minimized by equation (2):

$$0 = \frac{\partial E_{id}}{\partial \hat{c}_j} = \sum_k \sum_i \left[\lambda_{i,J,k} \left(\sum_j \hat{c}_j a_{i,j,k} \right) - h_{i,k}^{\lambda_k} \right]$$

or:

$$\sum_j \hat{c}_j \sum_k \sum_i \left[a_{i,J,k} a_{i,j,k} \right] = \sum_i \sum_i a_{i,J,k} h_{i,k}^{\lambda_k} \quad \text{for} \quad \forall J \in \left\{1,2,\ldots,n_j\right\} \qquad (3)$$

$$\text{If } \beta_{s,y} = \sum_k \sum_i \left[a_{i,s,k} a_{i,t,k} \right] \qquad (4)$$

$$\text{and } \lambda_j = \sum_k \sum_i a_{i,j,k} h_{i,k}^{\lambda_k} \qquad (5)$$

then by appropriate substitution into equation (3), we have:

$$\sum_j \hat{c}_j \beta_{J,j} = \gamma_j; \quad \forall J \in \left\{1,2,\ldots,n_j\right\} \qquad (6)$$

or, in the now familiar vector notation,

$$\vec{\hat{c}}[\beta] = \vec{\gamma}\,. \qquad (7)$$

This fortunate notation makes the explicit solution to the minimization of (1) obvious:

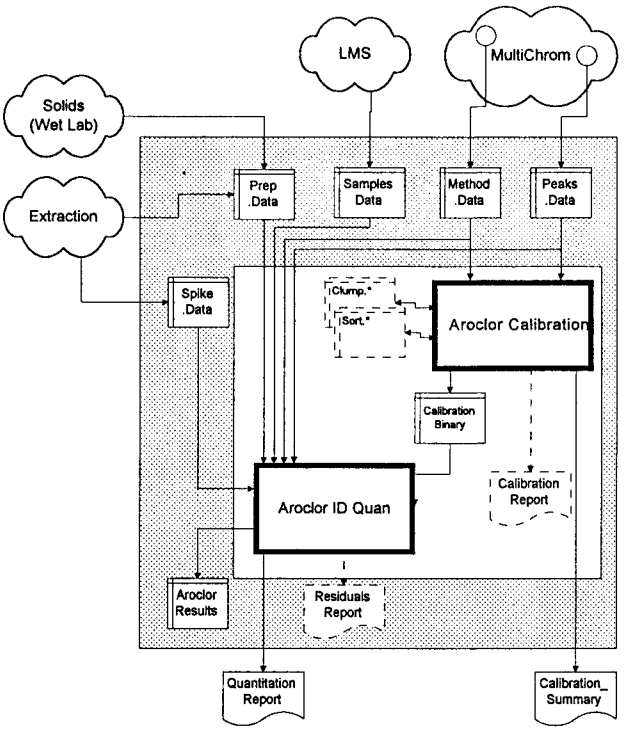
$$\vec{\hat{c}} = \vec{\gamma}[\beta]^{-1} \qquad (8)$$

where $[\beta]$ is strictly a function of the $a_{i,j,k}$ and $\vec{\gamma}$ is readily computed from the unknown chromatogram using λ_k and the $a_{i,j,k}$. Since the null-space solution $\vec{c} = (0,0,\ldots 0)$ satisfies the constraint, we are assured of the existence of a solution. Inspection of the 2^{n_k} solutions of equation (8) which satisfy $\hat{c}_j \geq 0, \forall_j$ yields the constrained solution to equation (2).

Figure 1 is a flow schematic for the Aroclor processing system. The system takes advantage of all peak data on both columns and utilizes peak processing and quantitation models to match linear combination assumptions. The system requires standardized input to provide objective, reproducible output. In practice, the first step in these analyses is to acquire calibration data for five levels of Aroclors 1221, 1232, 1016, 1242, 1248, 1254, 1260, 1262 and 1268. These are acquired on dual capillary columns and a total of 90 chromatograms are obtained in the calibration process. In the current setup, there are nine standard Aroclors under consideration and most give forty or more peaks on each column. Data are collected and stored using a Digital Equipment Corporation VAX minicomputer and a VG Multichrom chromatography data system. Although a large amount of data are obtained, no manual intervention is required and calibration is reduced to instrument and computer time. The VAX based Multichrom system watches the GC as a background processing job and captures files when available. The analyst is required to select files to be used in the calibration process and then process the chromatograms and quantitation reports using normal Multichrom data processing routines. These conventional reports are supplied in a data package presented with the analytical work. Raw electronic Multichrom data are also used directly to feed the calibration and ID/Quan routines which are modules in the PCB data processor. LIMS, and sample preparation data are electronically fed to the data processor for final concentration calculations and final report generation. Two important outputs come from the data processor. One is a quantitation report and the other is a calibration summary. These will be discussed in detail below.

Two modules are imbedded in the PCB Data processor. The first is the Aroclor Calibration module. Figure 2 is an outline of the module. This module reads

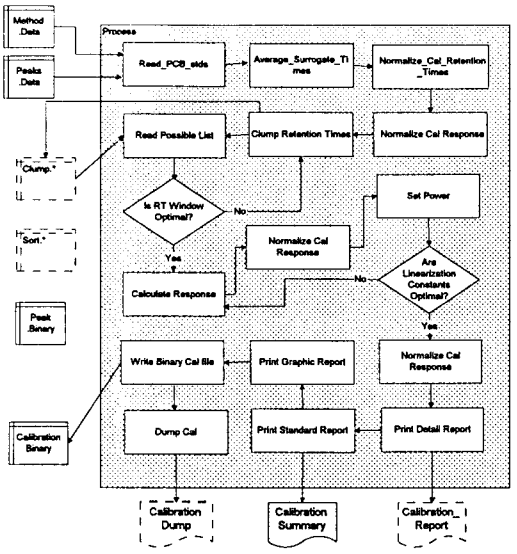
Figure 1-Overview of Aroclor System



packaged method and peak data from the Multichrom system and selects data from PCB standards. Retention time markers for all standards are averaged and all the peak data are normalized to these retention times. Useful RT markers are tetrachloro-m-xylene and the uncommon PCB congener, BZ#192. Heights for each peak are normalized by the respective standard concentrations and the retention times of all the peaks in all the standards are assigned to discrete retention time windows. This assignment is designated as clumping. Retention time windows are adjusted repetitively and calibration peaks are tested to find an optimum set of windows giving the maximum number of standardization peaks. These peaks must exhibit hits for all five levels of each standard on a column and have a normalized response variability of less than 15%. Many interferences and minor peaks are rejected at this step. If in the calibration process they do not meet these criteria, their windows will not be included but even a single peak on one Aroclor will be considered in setting the window if another Aroclor has all five valid peaks in that window.

The raw response of the electron capture detectors used to acquire GC data are approximately, but not truly linear. Detector electronics are used to linearize the signal but they become less effective as the detector ages. In other analytical schemes, linearity is often approximated by

Figure 2-Overview of Aroclor Calibration Module



narrowing the range of use. This severely attenuates the total capability of the detector and results in increased sample dilution and reanalysis. Without a linearity approximation, it becomes difficult to manually compute concentrations of linear combinations of standards based on non-linear signals. Power function linearization of the PCB response greatly improves the calibration model. Once raw peaks are classified for each of the five levels of a calibration sequence, the signal for each detector is linearized and evaluated by minimizing calibration variability through adjustment of the power parameter λ from 1 to λ. The linearization model is presented using equation (9).

$$h_i^{'} = h_i^{\lambda} \qquad (9)$$

where $h_i^{'}$ is the linearized height of peak i and h_i is the raw height of peak i. The linearization factor is reported for each detector for each calibration. It does not vary much and is between 1.0 and 1.2 for PCB work on all our systems.

Table 1 Example Page of a Calibration Report for One Column

8080_TERC Calibration Summary RTX-5

Calibration of 22-Jun-1995 19:23 through 23-Jun-1995 15:39 TMR - CE2745

2745:[062295] 01,02 22JUN951648

METHOD: 8080_STRAT:A

Peak Response Factors and Relative Deviations

Peak #	RT	Aroclor 1221		Aroclor 1232		Aroclor 1016	Aroclor 1242		Aroclor 1248		Aroclor 1254		Aroclor 1260		Aroclor 1262		Aroclor 1268	
38	11.962	0.593		0.368			0.563		1.454	8.40%	20.878	9.70%	50.196	5.70%	37.471	6.10%	0.395	
39	12.057	0.333		1.831	0.16		3.7	12.00%	9.514	7.60%	23.032	5.10%	13.196	11.10%	5.578	13.50%	0.303	
40	12.152	0.423		0.631			0.433		0.643		6.971	8.30%	18.77	7.70%	12.283	10.30%	2.613	
41	12.204												8.938	12.40%	14.99	12.20%	1.219	
42	12.304										3.049		4.765	12.90%	4.587	16.0%		
43	12.375	0.489		0.467			1.01		2.582	6.70%	34.138	6.50%	47.721	4.90%	22.036	8.70%		
44	12.483												6.222	11.30%	9.191	10.40%	0.505	
45	12.512						0.165		0.337		2.736	7.30%						
46	12.606	0.33		0.256			0.142		0.263		1.888	7.90%	30.914	8.00%	53.093	4.90%	20.77	7.80%
47	12.683	0.108									1.407		15.997	8.10%	19.48	7.70%	0.936	23.0%
48	12.811						0.294		0.774		8.827	8.50%	3.538	10.50%				
49	12.867												5.371	8.50%	9.031	6.20%	0.726	
50	12.981	0.176		0.123					0.097		1.892	7.60%	24.419	7.60%	32.01	5.70%	1.005	25.0%
51	13.075	0.096									1.27		14.01	9.90%	15.552	9.20%		
52	13.113																26.422	7.10%
53	13.153						0.248		0.53		6.87	5.50%	10.29	8.30%				
54	13.241														4.613		6.777	7.30%
55	13.397										4.596	5.80%	67.659	2.10%	85.906	2.30%	9.229	10.40%
56	13.632												1.97	6.60%	4.981	5.20%	4.915	4.50%
57	13.941	0.119							0.174		3.481	6.60%	26.327	5.80%	19.705	5.90%		
58	14.071										0.134		13.795	8.50%	33.881	5.80%	112.62	5.20%
59	14.175										0.206		15.851	7.60%	33.463	5.90%	91.464	2.80%
60	14.749																77.856	1.10%
61	14.805												6.151	6.20%	10.326	5.50%		
62	14.92												0.456		1.35		17.431	6.00%
63	15.178												14.142	6.80%	25.673	4.90%	27.019	5.40%
64	16.084												4.048	5.80%	8.177	5.60%	224.95	9.90%
65	16.927												0.465		0.175		33.57	8.70%
	Total	58.1	5.40%	190.1	5.00%	275.2	6.60%	327.1	5.90%	379	7.00%	440.6	7.20%	541.6	6.90%	535.2	5.80%	666	6.60%

Retention Time Marker/Surrogate Standard Summary

Compound	Mean RT	RT Dev. Range	RT Crit.	Mean RF	Recovery Range	Surr Crit
tetrachloro-meta-xylene	7.6983	-.0139 to .0106	Passes	1478.773	67.6 to 119.2%	Fails *
BZ#192 (heptachlorobiphenyl)	13.3127	-.0171 to .0140	Passes	813.101	94.6 to 110.2%	Passes

Notes:

1) Retention times linearly adjusted to place tetrachloro-meta-xylene at 7.6983 and BZ#192 (2,3,3',4,5,5',6-heptachlorobiphenyl) at 13.3127.

2) Peaks chosen within window of +/- 0.01647 of the mean RT of corresponding peaks in all mixtures.

3) Quantitation is based on HEIGHTS; instrument response is linearized by RESPONSE = HEIGHT^ 1.00486. &s-4R

Unverified(for laboratory use only) (HGJSTAR)21-JUL-95 07:12:08 8080_TERC_CALIBRATION 1.0-18(2MAR95 1342)

Table 2 Page 2 of a Calibration Detail Report for One Column

Aroclor Calibration Detail **Aroclor 1260 - RTX-35**

Calibration of 23-May-1995 13:07 through 24-May-1995 03:45 LJC - HP0850

MCD1:[8080] 13,14 23MAY951148

Peak Correspondence Listing - ALL Observed Peaks

	Concentration:50. ppb Acq'd:24-May-1995 02:02				Concentration: 100. ppb Acq'd:24-May-1995 02:28				Concentration: 200. ppb Acq'd:24-May-1995 02:53				Concentration: 400. ppb Acq'd:24-May-1995 03:19				Concentration: 800. ppb Acq'd:24-May-1995 03:45						
RT*	RT	EIGHT	err	nrmlzd	RT	EIGHT	err	nrmlzd	RT	EIGHT	err	nrmlzd	RT	EIGHT	err	nrmlzd	RT	EIGHT	err	nrmlzd	*	RF	%RSD
12.403	12.398	388	8	1016	12.396	687	1	1970	12.391	1148	-5	3576	12.4	2177	-2	7519	12.391	3944	6	14991	+	18.76	2.90%
12.624	12.618	2235	7	7749	12.613	4089	-1	15632	12.611	7079	-5	29563	12.62	12887	-3	59281	12.611	22763	6	114769	+	146.45	2.89%
12.724	12.718	1180	7	3691	12.713	2172	-1	7496	12.711	3710	-5	13960	12.72	6834	-3	28379	12.711	11866	6	53864	+	69.28	3.77%
12.927	12.922	4103	9	15692	12.918	7481	0	31522	12.913	12805	-6	58841	12.922	23131	-4	116927	12.913	40363	5	223200	+	287.86	4.27%
12.996					13.007	1586	19	5203	13.002	2718	13	9726	13.013	5017	18	19821					-	(49.64	2.60)
	13.009	857		2546													13.004	8825		38190	M		
13.14	13.133	5005	6	19765	13.129	9038	-2	39263	13.124	15394	-8	72870	13.136	27798	-4	144746	13.127	48413	5	275685	+	356.34	4.67%
13.233	13.229	358	9	923	13.224	615	0	1733	13.22	1010	-5	3080	13.231	1829	-1	6140	13.224	3276	10	12083	+	15.46	5.54%
	13.358	190		443	13.353	230		553	13.32	273		674	13.329	421		1114	13.32	704		2028	C		
13.493	13.484	2177	4	7516	13.48	3898	-4	14788	13.478	6630	-7	27401	13.489	12426	-3	56827	13.48	22418	5	112753	+	141.47	2.23%
13.654	13.649	1732	8	5764	13.644	3194	0	11734	13.64	5497	-5	22039	13.651	10085	-2	44592	13.642	17796	7	86238	+	109.91	2.78%
13.822	13.813	2328	5	8126	13.807	4355	-6	16817	13.804	7549	-9	31856	13.818	13992	-3	65220	13.807	24970	4	127791	+	161.17	1.65%
13.899	13.889	5250	3	20893	13.884	8615	-5	37137	13.882	14034	-8	65448	13.896	25737	-2	132357	13.887	45767	7	258265	+	331.68	6.61%
14.006	14	3037	8	11065	13.993	5602	-3	22531	13.991	9539	-6	41801	14.002	17343	-3	83690	13.993	30413	7	160674	+	206.3	3.80%
14.124	14.118	1650	7	5449	14.111	3098	-3	11326	14.109	5399	-6	21586	14.122	10060	-1	44466	14.111	17937	6	87029	+	109.58	1.52%
	14.329	2792		10037	14.322	1466		4749	14.322	2434		8556	14.336	3043		11092	14.324	1140		3546	C		
14.415	14.407	500	4	1361	14.402	921	-4	2768	14.4	1670	-6	5527	14.416	3204	0	11775	14.404	5870	8	23787	+	29.17	3.32%
14.705	14.698	3004	6	10927	14.691	5656	-4	22783	14.691	9824	-4	43254	14.704	18309	0	89126	14.691	32857	5	175766	+	220.55	1.40%
14.891	14.878	2047	0	6999	14.871	4008	-10	15273	14.869	7202	-12	30164	14.884	13577	-6	62982	14.871	24190	0	123168	+	153.93	2.39%
15.066	15.056	6047	3	24621	15.049	11244	-8	50595	15.049	19594	-7	96430	15.062	36888	-3	201046	15.049	66300	2	397173	+	496.69	1.47%
15.217	15.211	152	7	342	15.202	272	-6	673	15.202	501	-5	1364	15.218	1000	0	3045	15.204	1820	6	6107	+	7.44	5.22%
15.676	15.669	188	5	438	15.662	394	-5	1033	15.662	652	-4	1853	15.678	1243	1	3920	15.662	2298	5	8004	+	9.84	3.78%
15.833	15.824	122	4	264	15.813	141	-10	312	15.82	296	-2	741	15.838	633	4	1792	15.82	1157	6	3607	+	4.33	11.44%
16.009	16	1538	3	5021	15.996	2940	-4	10657	15.996	5109	-3	20247	16.011	9580	1	42011	15.996	17068	5	82155	+	103.28	1.70%
16.15	16.138	3117	0	11406	16.131	5921	-9	24027	16.133	10409	-6	46260	16.149	19599	-1	96460	16.131	35766	1	193958	+	240.07	1.96%
16.536	16.529	92	5	190	16.522	194	-5	452	16.52	370	-5	961	16.54	744	3	2162	16.52	1393	3	4476	+	5.32	9.17%
	16.693	83		170									16.709	85		174					B		
16.984	16.971	118	0	255	16.969	200	-5	470	16.969	376	-3	978	16.989	771	4	2251	16.971	1444	7	4667	+	5.56	7.84%
17.315	17.296	90	-6	185	17.3	112	-5	240	17.304	174	0	400	17.322	352	6	905	17.302	634	7	1796	+	2.28	13.59%
17.454	17.442	579	1	1614	17.438	1081	-6	3336	17.44	1942	-2	6586	17.46	3763	5	14192	17.438	6790	4	28169	+	34.77	3.21%
17.657	17.647	1352	2	4322	17.642	2586	-5	9181	17.644	4562	0	17750	17.662	8740	4	37765	17.638	15535	1	73648	+	92.04	2.39%
	17.956	301		755	17.956	262		644	17.953	210		496	17.976	182		422	17.951	183		425	C		
18.967	18.956	432	0	1151	18.956	840	-2	2487	18.953	1551	0	5072	18.973	3111	5	11380	18.947	5559	0	22327	+	27.37	5.76%
	19.42	267		658	19.424	257		629	19.42	281		697	19.44	282		699	19.416	193		450	C		
									19.924	216		515	19.942	198		465					B		
	20.133	982		2984	20.142	896		2681													B		
	20.273	5526		22177	20.276	9535		41781	20.271	16110		76821	20.287	29942		157791	20.264	51165		293959	R		
Total:		136642		594845		183145		794558		264706		1E+06		465193		2E+06		751707		4E+06		3645.5	3.19%

Notes:

1) Retention times linearly adjusted to place tetrachloro-meta-xylene at 6.5481 and decachlorobiphenyl at 16.9978.
2) Peaks chosen within window of +/- 00.01981 of the mean RT of corresponding peaks in all mixtures.
3) Quantitation is based on HEIGHTS; instrument response is linearized by RESPONSE= HEIGHT^ 1.13081.

AROCLOR_CALIBRATION 5.2-3(12AUG93 1142) (SJF)26-MAY-95 16:17:11

Once calibration peak windows are selected for each Aroclor and the responses linearized, normalized calibration response factors are computed for each five peak group for each clump for each Aroclor using least squares analysis with errors weighted by concentration as presented in equation 10.

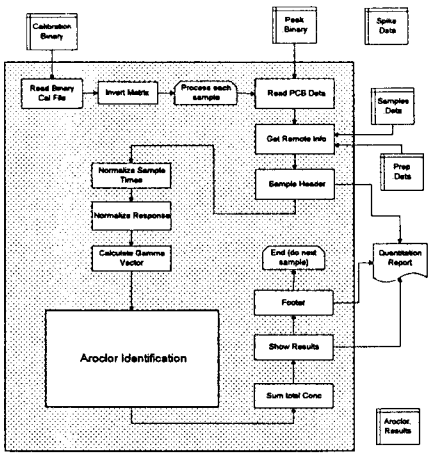
$$RF_{ij} = \sum_{l} h_i / \sum_{l} c_{ij} \qquad (10)$$

where $h_i^{'}$ is the linearized height of peak i for a given Aroclor (j) and (c_{ij}) is the lth concentration of the Aroclor.

Retention times, window widths, normalization constants and response factors are made available to the identification/quantification program in a calibration binary file and summarized in a calibration summary report. The last page of a calibration report showing an example of information available is presented in (table 1). The table presented is for one column (RTX-5) and shows for each Aroclor the weighted response factor in the units of height/concentration and the percent relative deviations of the actual response factors vs. the predicted response factors.

A detailed calibration report is also available. This report is quite long and an example page for part of the Aroclor 1260 listing is presented in table 2. The column RT* is the mean retention time for a window of ($\pm 0.01981 \times$ RT*) in which a PCB peak can be found. The RT column under each concentration of Aroclor 1260 shows the actual time of a peak found in the window and the err is the time difference in milliminutes between RT* and RT. The height of each peak before linearization is itemized and the height after application of the linearization is given under the heading nrmlzd. There is a peak status summary

Figure 3-Overview of Aroclor ID Quan Module



under the column headed by *. The designation + indicates a good group of five peaks in the retention time window meeting calibration acceptance criteria. The designation - indicates a group of peaks that do not meet calibration acceptance criteria, but correspond to a group which meets such criteria in another Aroclor. These peaks are not rejected and are used to estimate the 1260 response factor for this peak. C indicates a peak set that fails %RSD criteria which may be from a contaminant. M indicates peak sets which are incomplete. B indicates peak sets that fail both calibration criteria (%RSD >15% and less than five peaks per Aroclor). C, M and B peak sets are not used. R indicates a peak set is from a retention time marker. RF and % RSD are from the calibration summary.

The second module imbedded in the Process PCB Data processor is the Aroclor ID Quan module. Figure 3 is an outline of the module. The process starts with a processing of each chromatogram pair. Peak data are read from the

Figure 4-Aroclor Quantitation Report

8080 TERC Quantitation Report

METHOD: 8080_STRAT:A
Based on Calibration of 22-Jun-1995 19:23 through 23-Jun-1995 15:39.
2745:[062295] 01,02 22JUN951646

AR1242 100ppb
Acquired:23-Jun-1995 02:45
01,02 22JUN951646,20,1
TMR - CE2745

AROCLOR Concentration Estimate (ppb)

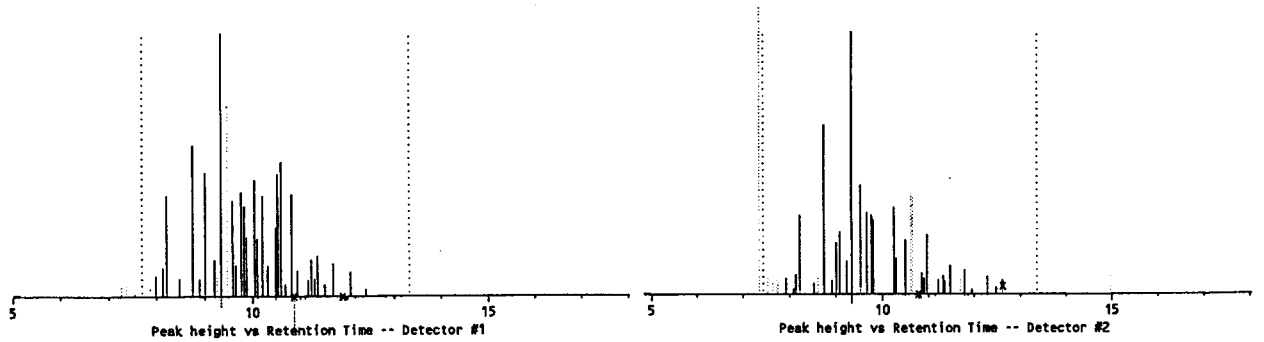
#Comp	1221	1232	1016	1242	1248	1254	1260	1262	1268	Fit	Imp	SS	MS	df	Ratio	F
0	----	----	----	----	----	----	----	----	----	0	0	0.144	0.003	50	1.0000	0.5000
1	----	----	----	107.4	----	----	----	----	----	996	1000	0.000	0.000	49	0.0023	0.0000
2	----	----	----	103.9	4.54	----	----	----	----	997	784	0.000	0.000	48	0.7972	0.2161
3	----	5.78	----	100.1	5.30	----	----	----	----	997	560	0.000	0.000	47	0.9570	0.4397
4	----	5.87	1.37	98.1	5.95	----	----	----	----	997	540	0.000	0.000	46	0.9716	0.4601
5	1.48	3.91	1.54	98.7	6.07	----	----	----	----	997	512	0.000	0.000	45	0.9918	0.4875

Estimated composition of injected extract (Fit is Extremely good ; IFIT=997):
103.9 ppb of Aroclor 1242 + 4.5 ppb of Aroclor 1248

Outlier (Ignored) Peaks

Peak	SS	Peak	SS
non-PCBs	0.5146	TCX	0.2739
BZ192	0.1240	1- 25	0.0003
2- 46	0.0000	2- 21	0.0000
1- 37	0.0000		

RT Marker / Surrogate	RT-Det#1	RT-Det#2	RT (QC)	Recovery-Det#1	Recovery-Det#2
tetrachloro-meta-xylene	7.698(-0.001)	7.444(+0.012)	Passes...	7.813 ug/l	8.228 ug/l
BZ#192 (heptachlorobiphenyl)	13.311(-0.002)	13.367(+0.009)	Passes...	10.182 ug/l	9.782 ug/l



Sample is Aroclor 1242 Initial Calibration Standard at 100ppb
Extract undiluted for this injection.

Aroclor Concentrations
in undiluted extract (ug/l)

Aroclor 1221	50	U
Aroclor 1232	50	U
Aroclor 1016	50	U
Aroclor 1242	100	
Aroclor 1248	50	U
Aroclor 1254	50	U
Aroclor 1260	50	U
Aroclor 1262	50	U
Aroclor 1268	50	U

U - Less than indicated concentration

KEY:
Surrogate/RT Marker
non-PCB Peak (outside window)
PCB Peak
Estimation error (Peak - estimate)
Outlier Peak (treated as non-PCB)
Peaks normalized to maximum of detected PCBs; higher peaks and large overestimates truncated.

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peak binary and linked with prep data and samples data. A sample header is generated for a two column report.

Retention times and responses are normalized and the data are processed through the Aroclor identification routines using the minimization procedures for goodness-of fit and calibration data from the calibration binary. Results are totaled, stamped with time and process information, and sent to the two column report. The process is repeated for the next sample. An example of a two column report for Aroclor 100 ppb 1242 processed as a sample is shown in Figure 4.

If extraction information is available to the module, results will be reported in terms of original sample concentration along with a summary of dilution status and sample weights, moistures, etc. Sample and file identifications for both columns are in the header. Surrogate data are summarized and a table showing possible Aroclor mixture combinations accounting for the pattern are tabulated along with a goodness-of-fit index and improvement index for adding additional Aroclors to the solution (based on 1000). Additional statistical parameters and outlier peak lists are also tabulated.

The results chosen by the program are summarized below the table and indicate that the most significant improvement in fit was obtained with a mixture of 103.9 ppb Aroclor 1242 and 4.5 ppb Aroclor 1248. Rounded concentrations are summarized in a "Form 1" fashion at the bottom of the report.

The graphical display shows peak and fit data for both columns. A key is provided for the display. Surrogate/retention time marker peaks are indicated by wide-spaced dotted lines. Non-PCB peaks (or peaks outside windows) are indicated by finely dotted lines. Peaks with an X are in windows but considered outliers by the program. These may be coeluting interferences and are not used in the quantitation. Solid lines are PCB peaks. Starting from the top of each peak, they remain solid to zero if they are perfectly accounted for by back fitting the Aroclor standard response for the peak at the stated concentration of the quantitation report. The line is partially dashed to zero if the corresponding peak in a standard does not quite account for the sample peak. It is dashed below zero if the concentration of a standard peak over estimates the sample peak.

Data Validation

The software is extensively tested and documented. The revision level of the software used to process a group of samples (along with the revision date) are stamped at the bottom of each quantitation report. Nevertheless, it is necessary to determine if the system is producing valid, meaningful data for the intended use. This is normally done during data validation.

The essence of data validation of results generated by this process can be stated in terms of whether or not the process has been tested to ensure its ability to meet the data quality objectives of a program for which it is intended. Assurance of the program's ability to meet the analytical DQOs is provided by testing a range of known mixtures and evaluating their results proir to a project. Once the processing systems have been demonstrated to meet the DQOs, audit trails must be placed on the program files to ensure they do not change from their tested state.

In general, numerical techniques on large amounts of data are nearly impossible to reproduce without a full data processing system. This causes complications in data validation when validators are faced with the challenge of checking results with limited resources. It is general policy to check a set of calculations during the validation process by recalculation to see if the exact answer can be reproduced. This approach must be modified to validate results from the data processor. There is an advantage in that the results are not subject to human intervention and are more likely self consistent than individually hand calculated results. This allows for a better basis in looking at a subset of the data to test each functional process.

The routine produces a number of displays to assist in validation. The first is a calibration report which gives peak windows determined for each standard. The raw chromatograms are supplied and the task for the validator becomes one of not determining how the program classified the peaks but whether the validator agrees with the result based on the raw data. Randomly selected peaks can be readily evaluated.

Linearized responses are used by the program. Raw responses are included in the data package along with all chromatograms and the computer generated power factor (on the calibration report summaries). Again, the task is not to determine how the program came up with the best factor but to evaluate randomly selected data points to see if they are adequately linearized by the model.

The calibration is evaluated by computer generated reports of a back calculation of each calibration and periodic check standard as unknowns using the same quantitative routines applied to the associated unknowns. These are included in the data package.

The ability of the program to evaluate mixtures is evaluated by running a complex mixed standard as an unknown through the entire process. Subtle or unexpected changes in the program behavior can be detected by evaluation of these mixtures.

Each of the original chromatograms is presented for the standards and samples in the data package. Using manual pattern matching it is possible for the validator to see if the identity of unknowns is best explained by the standards. This is what is customarily done for any PCB data validation and the quantitation report display is available to assist the validator in determining how well an identity and quantification accounts for the unknown chromatogram.

Conclusions

The numerical techniques used give objective interpretation of the best combination of Aroclors to account for an unknown PCB pattern and also allow full utilization of electron capture detector signal range. They take advantage of all useful signals and have a high resistance to bias from interference. They allow for automated processing of large amounts of data and a full calibration scheme allows for identification of any combination of Aroclors found in the unknowns without assuming a composition in advance of the analysis. Restrictions based on the inability to deal with all the data are eliminated. Instead of physically making a standard mixture to best match the sample before quantitation, the mix is effectively prepared from mathematical modeling each of the individual Aroclor standards.

This technique is especially effective in programs targeting large numbers of samples and there can be substantial benefits in uniform analytical interpretation along with significant long term cost savings in data validation.

References

(1) Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, SW-846 3rd Edition, Final Update 1 (1992)

Figure 1-Overview of Aroclor System

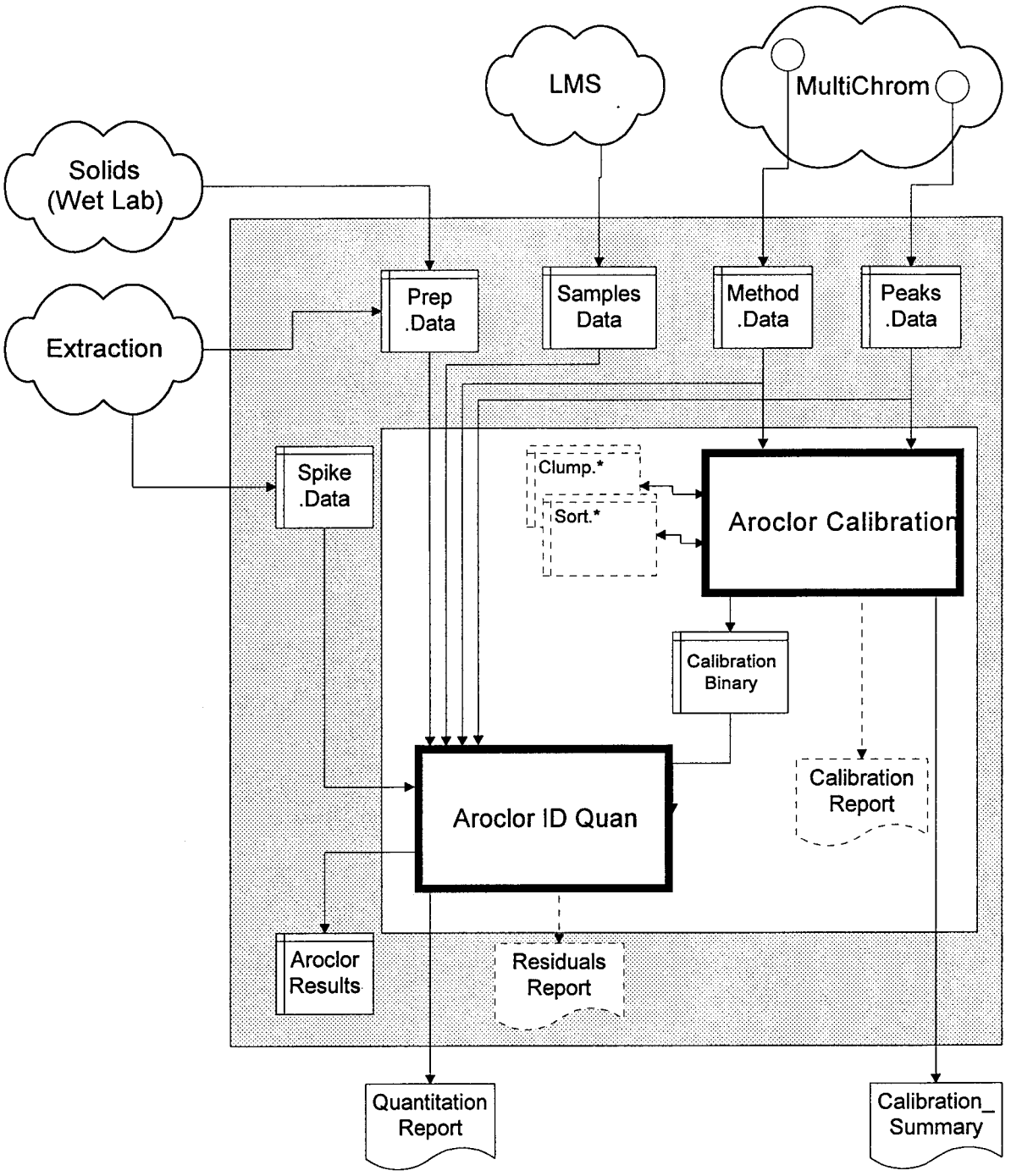


Figure 2-Overview of Aroclor Calibration Module

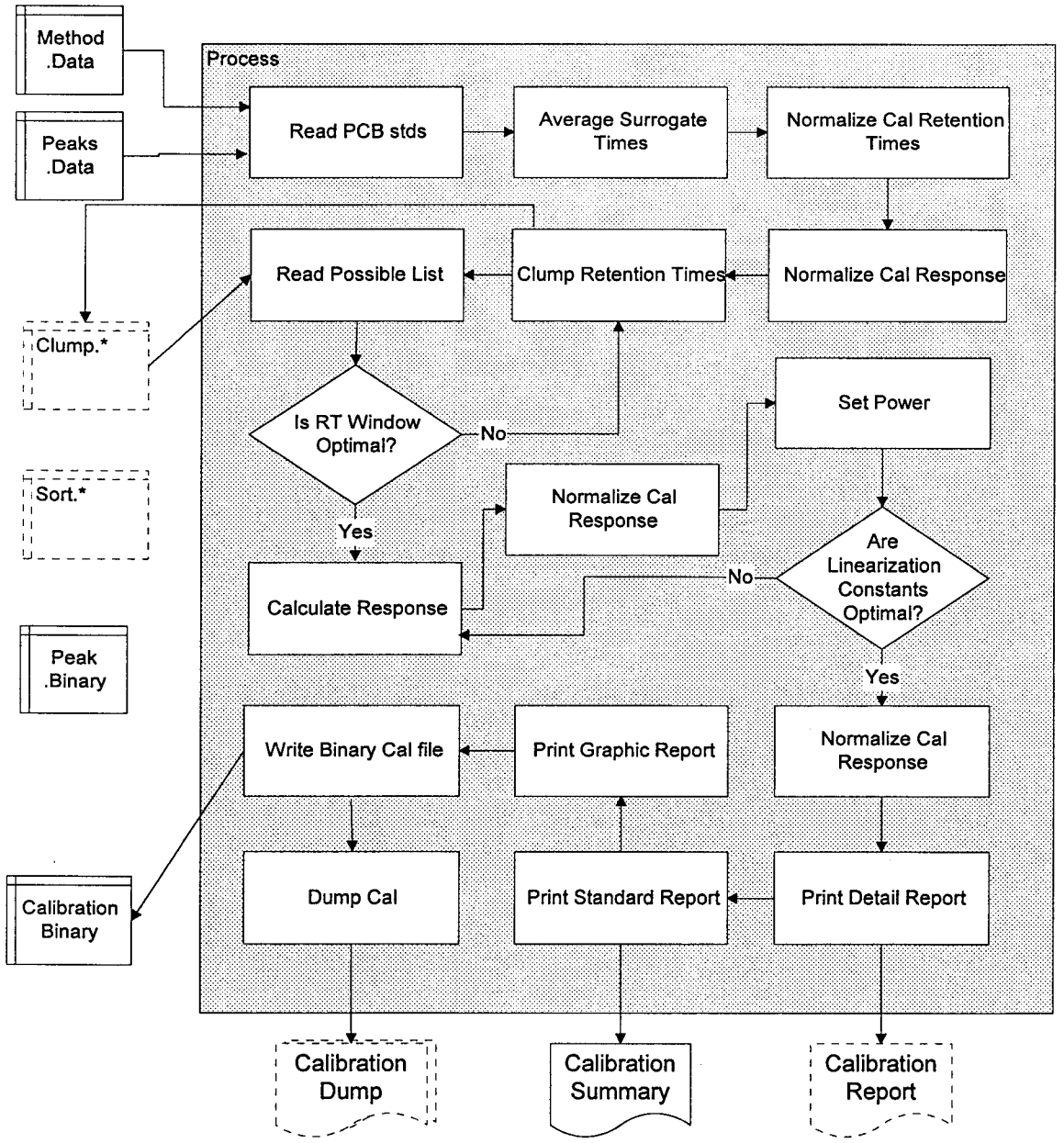


Figure 3-Overview of Aroclor ID Quan Module

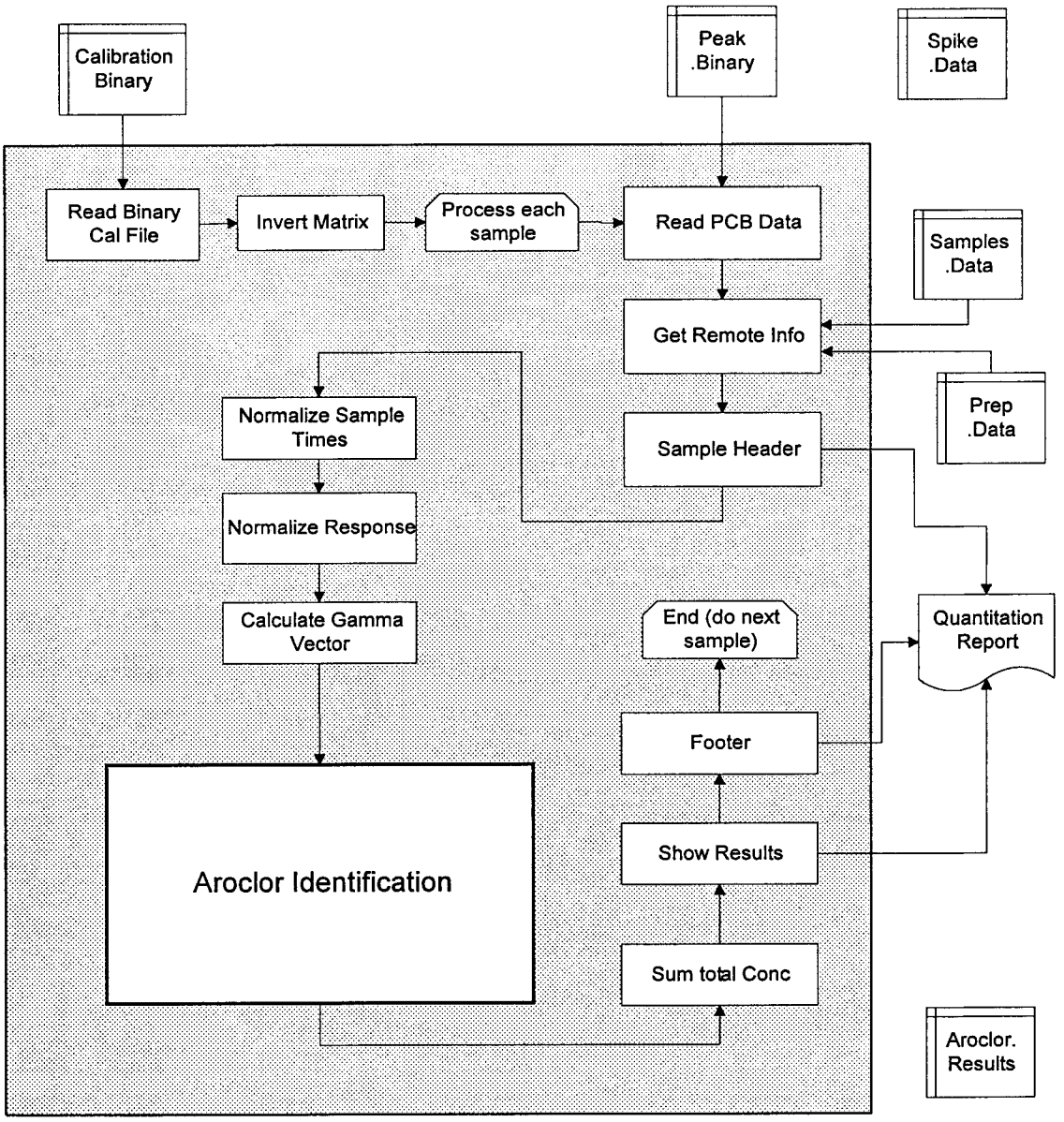


Figure 4-Aroclor Quantitation Report

8080 TERC Quantitation Report

METHOD: 8080_STRAT:A
Based on Calibration of 22-Jun-1995 19:23 through 23-Jun-1995 15:39.
2745:[062295] 01,02 22JUN951646

AR1242 100ppb
Acquired:23-Jun-1995 02:45
01,02 22JUN951646,20,1
TMR - CE2745

AROCLOR Concentration Estimate (ppb)

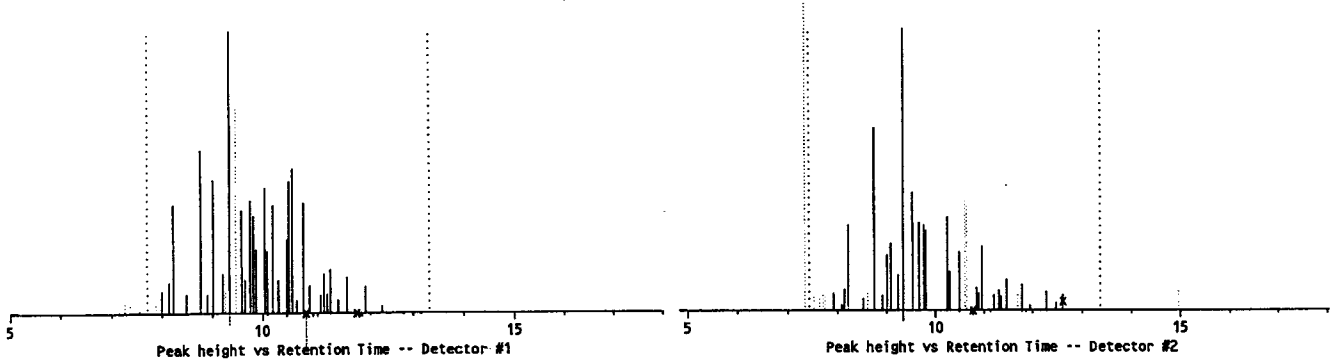
#Comp	1221	1232	1016	1242	1248	1254	1260	1262	1268	Fit	Imp	SS	MS	df	Ratio	F
0	----	----	----	----	----	----	----	----	----	0	0	0.144	0.003	50	1.0000	0.5000
1	----	----	----	107.4	----	----	----	----	----	996	1000	0.000	0.000	49	0.0023	0.0000
2	----	----	----	103.9	4.54	----	----	----	----	997	784	0.000	0.000	48	0.7972	0.2161
3	----	5.78	----	100.1	5.30	----	----	----	----	997	560	0.000	0.000	47	0.9570	0.4397
4	----	5.87	1.37	98.1	5.95	----	----	----	----	997	540	0.000	0.000	46	0.9716	0.4601
5	1.48	3.91	1.54	98.7	6.07	----	----	----	----	997	512	0.000	0.000	45	0.9918	0.4875

Estimated composition of injected extract (Fit is Extremely good ; IFIT=997):
103.9 ppb of Aroclor 1242 + 4.5 ppb of Aroclor 1248

Outlier (Ignored) Peaks

Peak	SS	Peak	SS
non-PCBs	0.5146	TCX	0.2739
BZ192	0.1240	1- 25	0.0003
2- 46	0.0000	2- 21	0.0000
1- 37	0.0000		

RT Marker / Surrogate	RT-Det#1	RT-Det#2	RT (QC)	Recovery-Det#1	Recovery-Det#2
tetrachloro-meta-xylene	7.698(-0.001)	7.444(+0.012)	Passes...	7.813 ug/l	8.228 ug/l
BZ#192 (heptachlorobiphenyl)	13.311(-0.002)	13.367(+0.009)	Passes...	10.182 ug/l	9.782 ug/l



Sample is Aroclor 1242 Initial Calibration Standard at 100ppb
Extract undiluted for this injection.

Aroclor Concentrations
in undiluted extract (ug/l)

Aroclor 1221	50	U
Aroclor 1232	50	U
Aroclor 1016	50	U
Aroclor 1242	100	
Aroclor 1248	50	U
Aroclor 1254	50	U
Aroclor 1260	50	U
Aroclor 1262	50	U
Aroclor 1268	50	U

U - Less than indicated concentration

KEY:
- ····· Surrogate/RT Marker
- ····· non-PCB Peak (outside window)
- ——— PCB Peak
- ——— Estimation error (Peak - estimate)
- x····· Outlier Peak (treated as non-PCB)

Peaks normalized to maximum of detected PCBs;
higher peaks and large overestimates truncated.

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SEVERN TRENT STL

Project Memorandum

Date: June 26, 2006

From: Jim Madison

Subject: Kalamazoo Project PCB Procedure

This Project Memorandum serves to document the analytical requirements for the analysis of samples for PCBs from the Kalamazoo site. All procedures follow those outlined in SOP No. LM-GC8082, Revision 7, Effective 12/12/05, with the following clarifications:

Section 4.3 All water samples are subjected to sulfuric acid cleanup. All solid matrix samples are subjected to GPC, Silica Gel, Sulfuric Acid and Sulfur cleanups.

Section 9.5 Five-point ICAL standards are analyzed for every Aroclor, rather than just for Aroclors 1016 and 1260 (referred to as AR1660). Additionally, rather than a single AR1660 standard as a CCV every ten samples, analyze two Aroclor standards every ten samples – using Aroclor 1248 plus alternating standards for Aroclors 1242, 1254 and 1660.

Section 10.2 Invoke the five-point ICAL standards for each Aroclor.

Section 10.4 Retention time windows are calculated by STL's PCB software algorithm. The surrogates must elute within 0.04 minutes of their mean elution time from the initial calibration for the PCB software algorithm to recognize each Aroclor.

Section 10.5 A stand-alone second source ICV standard is not analyzed after the initial calibration.

Section 10.6 Analyze two CCV Aroclor standards every ten samples – using Aroclor 1248 plus alternating standards for Aroclors 1242, 1254 and 1660.

Section 11.2 Include the five-point calibration for each Aroclor, plus the double alternating CCVs, in the analytical sequence.

Section 11.5 Aroclor identification and quantification is accomplished using STL's PCB software algorithm.

Section 13 Aroclor identification and quantification is accomplished using STL's PCB software algorithm.

Table 1 Reporting Limits for water samples are 0.05 ug/L for each Aroclor. Solid reporting limits are 50 ug/Kg for each Aroclor. Control limits are project specific.

Project Manager Approval Jim Madison 6/26/2006

Organic Section Head Approval 6/26/06

ARCADIS BBL

Attachment 1-3

STL SOM Organic SOPs

STL BURLINGTON
STANDARD OPERATING PROCEDURE

ANALYTICAL METHOD FOR LOW/MEDIUM VOLATILES
CLP SOW SOM01.0 / SOM01.1

Applicable Matrix: Water and Soil/Sediment

APPROVAL SIGNATURES

William S. Cicero
Laboratory Director
Date: January 22, 2007

Kirstin L. McCracken
Quality Assurance Manager
Date: January 22, 2007

Mark T. Phillips
Department Manager
Date: January 22, 2007

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the analysis of water, soil and sediment samples samples for the volatile organic compounds listed in Section 18.0, Table 1. This procedure is specific to the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) SOM01.1.

1.2 The EPA has identified and published in the SOW the following problems associated with some compounds analyzed by this method:

- Chloromethane, vinyl chloride, bromomethane, and chloroethane can display peak broadening if the compounds are not delivered to the GC column in a tight band.
- Acetone, hexanone, 2-butanone, and 4-methyl-2-pentanone have poor purge efficiencies.
- 1,1,1-Trichloroethane and all the dichloroethanes can dehydrohalogenate during storage or analysis.
- Chloromethane can be lost if the purge flow is to fast.
- Bromoform is one of the compounds most likely to be adversely affected by cold spots and/or active sites in the transfer lines. Response of its quantitation ion (m/z 173) is directly affected by the tuning of the GC/MS to meet the instrument performance criteria for 4-bromofluorobenzene (BFB) at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

2.0 SUMMARY OF METHOD

2.1 Water Samples

Deuterated monitoring compounds (DMCs) and internal standards are added to each sample and the entire vial is placed in the instrument autosampler. An inert gas is bubbled through 5 mL of sample in a purge and trap chamber maintained at ambient temperature. The purgeable compounds are transferred from the aqueous phase to the vapor phase and swept through a sorbent column where the purgeables are trapped. After purging is complete, the sorbent column is heated and backflushed with the inert gas and the purgeables are desorbed onto a GC column. The GC is temperature programmed to separate the purgeable compounds that are then detected with a mass spectrometer.

2.2 Low-Level Soil/Sediment Sample

Approximately 5 g of sample is analyzed in a pre-weighed 44 mL VOA vial with 5 mL of VOA-free reagent water. DMCs and internal standards are added to each sample and the entire vial is placed in the instrument autosampler. The sample vial is heated to 40°C and the volatiles are purged through a sorbent trap using an inert gas combined with

agitation of the sample. After purging is complete, the trap is heated and backflushed with the inert gas and the purgeables are desorbed onto a GC column. The GC is temperature programmed to separate the purgeable compounds that are then detected with a mass spectrometer.

2.3 Medium-Level Soil Sample

A sample weight of 5 g soil is collected and preserved in methanol. A 5 mL aliquot of the methanol preserved sample is added to VOA-free reagent water in a 44 mL VOA vial. DMCs and internal standards are added to each sample and then an inert gas is bubbled through the sample contained in a purge and trap chamber maintained at ambient temperature. The purgeable compounds are transferred from the aqueous phase to the vapor phase and swept through a sorbent column where the purgeables are trapped. After purging is complete, the sorbent column is heated and backflushed with the inert gas and the purgeables are desorbed onto a GC column. The GC is temperature programmed to separate the purgeable compounds that are then detected with a mass spectrometer.

2.4 This procedure is based on CLP SOW SOM01.0 Exhibit D, Trace Volatiles and subsequent change documents published by the EPA in May 2005 (SOM01.1).

The laboratory has implemented the following modifications from the reference test method:

- The laboratory uses a 5 mL purge volume instead of 25 mL.
- For 1,4-dioxane, the laboratory uses a minimum response factor of 0.002 instead of 0.005.

3.0 DEFINITIONS

3.1 See Appendix C for a list of definitions.

4.0 INTERFERENCES

4.1 Method interference may be caused by impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory method and instrument blanks. The use of non-Polytetrafluoroethylene (PTFE) tubing, non-PTFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during storage and handling. Therefore samples analyzed by this procedure must be stored separately from other laboratory samples and standards and must be analyzed in a room demonstrated to be free from contaminates.

4.3 Contamination by carryover can occur whenever high level and low level samples are

sequentially analyzed. To reduce carryover, the purging device and sampling syringe must be rinsed with reagent water between sample analyses. For samples containing large amounts of water-soluble materials, suspended solids, high-boiling compounds, or high purgeable levels, it may be necessary to wash out the purging device with a detergent solution between analyses, rinse it with distilled water, and then dry it in an oven at 105^{o}C. The trap and other parts of the system are also subjected to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4.4 The volatile laboratory and preparation area should be completely free of solvents, except for methanol.

5.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of each reagent used in this procedure has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Use reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Safety glasses, gloves and protective clothing must be worn.

The following analytes covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, vinyl chloride and 1,4-Dioxane.

5.2 Primary Materials Used

Table 2, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the Material Safety Data Sheet (MSDS). The table does not include all materials used in the procedure. A complete list of materials used can be found in Section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 QEC 44mL Borosilicate glass vials, Teflon-lined septa.

6.2 Syringes: 10 μL-2.5 mL Hamilton Gastight® syringes.

6.3 2 mL and 4 mL Borosilicate glass vials with Mininert® screw-cap on/off valves.

6.4 Autosampler - Tekmar ALS 2050 or Archon.

6.5 Purge and Trap Device - Tekmar LCS 2000 or Tekmar Velocity XPT.

Trap Specifications - 25 to 30cm x 2.5mm ID minimum, Supelco VOCARB 3000 (K-type trap), or equivalent.

6.6 GC System - Hewlett Packard 5890.

GC Columns - J&W DB624 75m x 0.53mm x 3.0 μm film (jet separator configuration).

6.7 MS System - Mass Spectrometer: Hewlett-Packard 5971.

The MS system must be capable of producing an averaged mass spectrum for the instrument performance check solution (25 ng/uL BFB) that meets the acceptance criteria detailed in Table 3.

6.8 Data System

Data Acquisition - ChemStation Version B.01.00

Data Reporting - Target Version 3.5

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Methanol (CH_3OH), purge & trap quality – OmniSolv high purity solvent.

VOA-Free Reagent Water: Boil water that has been filtered through the laboratory Milli Q Plus ™ filtration system for one hour then purge with helium for a minimum of one hour.

7.2 Standards

Prepare working standard solutions from stock standard solutions purchased from commercial vendors. See Appendix B for the recommended formulation for the preparation of the following standard solutions. Assign an expiration date to the prepared solutions as specified in the following section. Store the stock standards and primary working standard solutions, except the instrument performance check solution, protected from light at a temperature of -10^oC to -20^oC. Store the instrument performance check solution protected from light at a temperature of 4^oC ($\pm 2^oC$). Individual prepared calibration standards may be held for up to 24 hours after time of preparation if they are stored in Teflon sealed screw-cap vials with zero headspace at 4^oC ($\pm 2^oC$) and protected from light. Store all standards separately from samples and other chemical materials.

Instrument Performance Check Solution (BFB): The instrument performance check solution consists of 4-Bromofluorobenzene in methanol. Prepare the solution in a volumetric flask using the formulation specified in Appendix B. Assign an expiration date of 6 months from date made unless the expiration date of any parent component is sooner, in which case use the earliest expiration date.

SOM Trace Calibration Mix: The trace calibration mix contains the target and DMC compounds listed in Table 1, except for 1,4-dioxane and 1,4-dioxane-d_8, in methanol. Prepare the calibration mix in a 4 mL borosilicate glass vial with Mininert® valve and assign an expiration date of one month from date made unless the expiration date of any parent component is sooner, in which case use the earliest expiration date. Use the calibration mix to prepare the individual calibration standards and the opening and closing CCV standard.

SOM 1,4-Dioxane Calibration Mix: Prepare the 1,4-dioxane calibration mix in a 2 mL borosilicate glass vial with Mininert® valve. Assign an expiration date of one month from date made unless the expiration date of any parent component is sooner, in which case use the earliest expiration date. Use the 1,4 Dioxane calibration mix is used to prepare the individual initial calibration standards and opening and closing CCV.

SOM Internal Standard Spike Mix (ISTD): The ISTD contains 1,4-dichlorobenzene-d_4, chlorobenzene-d_5, and 1,4-difluorobenzene in methanol. Prepare the ISTD in a 4 mL borosilicate glass vial with Mininert® valve. Assign an expiration date of one month from date made unless the expiration date of any parent component is sooner, in which case use the earliest expiration date. Add the ISTD solution all calibration standards, blanks, and samples at a final concentration of 50 ug/L.

DMC Mix (SSTD): The SSTD contains all compounds listed in Table 4 in methanol. Prepare the SSTD in a borosilicate 4 mL glass vial with Mininert® valve. Assign an expiration date of one month from date made unless the expiration date of any parent component is sooner, in which case use the earliest expiration date. Add the SSTD to all blanks and samples at a concentration of 50 μg/L for each non-ketone DMCs, 100 μg/L for ketone DMCs, and 1250 ug/L1,4-dioxane-d_8.

Matrix Spike Mix (MS): The MS contains compounds listed in Table 5 in methanol. Prepare the MS in a 2 mL borosilicate glass vial with Mininert® valve. Assign an expiration date of one month from date made unless the expiration date of any parent component is sooner, in which case use the earliest expiration date. Add the MS spike to each MS/MSD samples at a concentration of 50 μg/L.

Initial Calibration and Continuing Calibration Verification (CCV) Standards: The initial calibration standards are prepared in 44 mL borosilicate glass vials using the SOM trace calibration mix and VOA-free reagent water. Prepare five initial calibration standards containing all of the target compounds and DMCs at the following concentrations: all non-Ketone targets and associated DMCs, except 1,4-Dioxane at 5.0, 10, 50, 100, and 200 μg/L; all ketones and associated DMCs at 10, 20, 100, 200, and 400 μg/L; and 1,4-Dioxane and its associated DMC 1,4-Dioxane-d_8 at 100, 200, 1250, 1000, and 2000

μg/L. Note that the m plus p-Xylene isomers total 5.0, 10, 50, 100, and 200 μg/L. Add ISTD to each calibration point at a concentration of 50 μg/L. See Appendix A for initial calibration formulation.

Prepare the opening and closing CCV at the mid-point concentration of the calibration in 44 mL borosilicate glass vials using the SOM trace calibration mix and VOA-free reagent water. See Appendix A for CCV formulations.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Sample Collection

The laboratory does not perform sample collection, thus these procedures are not applicable to this SOP. The sample collection procedure performed should be in accordance with the Analytical Method for the Analysis of Volatile Organic Compounds, Exhibit D, USEPA Contract Laboratory Program Statement of Work for Organic Analysis, (SOM01.1), May 2005. The laboratory provides per client request, pre-certified, pre-preserved sample collection containers and Encore sampling devices, which are shipped directly to the sampling site.

8.2 Sample Receipt & Preservation & Storage

Water
Water samples should be received in 44 mL VOA vials that were field preserved to a pH less than 2 at the time of collection. Upon laboratory receipt, store the water samples at a temperature of 4°C (±2°C) until time of analysis in a refrigerator dedicated to the storage of VOA samples. Store the samples with an aqueous storage blank which is subsequently analyzed to assess potential contamination that may have occurred during laboratory storage.

Soil/Sediment
Soil and sediment samples may be received from the field in pre-prepared closed system purge and trap vials, pre-weighed glass vials or in field core sampling containers.

Samples that are collected in pre-prepared closed system purge and trap vials may have been field preserved with sodium bisulfate or collected unpreserved in water. Samples that are received unpreserved in water must be analyzed within 24 hours or kept frozen at at a temperature of -7°C until time of analysis. Place samples that are to be stored at -7°C on their side prior to being frozen. Store samples that are field preserved with sodium bisulfate at a temperature of 4°C (±2°C) until time of analysis in a refrigerator dedicated to the storage of VOA samples.

Medium level soil/sediment samples may be received from the field in pre-weighed vials that contain methanol. Store the methanol preserved samples at a temperature of 4°C (±2°C) until time of analysis in a refrigerator dedicated to the storage of VOA samples.

For samples received in field core sampling containers, the samples must be transferred into pre-prepared closed system purge and trap vials within 24 hours of receipt or the

samples must be stored at -7°C. Place samples that are to be stored at -7°C on their side prior to being frozen.

Aqueous storage blanks must be stored with preserved low/medium soils and with water samples at a temperature of 4°C (±2°C). An inert sand storage blank must be stored with the unpreserved low/medium soil samples at a temperature of -7°C.

Unless otherwise specified by client or regulatory program, all samples are held from the time of receipt until 30 days after delivery of a complete, reconciled data package. After 30 days, any unused portions of the samples may be disposed of in a manner that complies with all applicable regulations.

8.3 Contract Required Holding Times

Analysis of water must be completed within 10 days of Validated Time of Sample Receipt (VTSR).

9.0 QUALITY CONTROL

9.1 Blanks: A method blank, storage blank and instrument blank(s) are prepared and analyzed to determine the level of contamination that may be associated with sample processing and analysis (method blank), sample storage (storage blank) or carryover from analysis of a previous sample (instrument blank). The frequency requirements for analysis and criteria are summarized as follows:

Method Blank: The method blank is analzyed at least once in every 12 hour time period on each instrument used for analysis. See Section 10.2 for a definition of the 12-hour time period. The method blank must be analyzed after the CCV and before analysis of any samples or storage blanks. If samples are analyzed in the same sequence as initial calibration, the method blank must be analyzed immediately after the calibration.

Storage Blank: The storage blank is analyzed after all samples in the SDG have been analyzed.

Instrument Blank: Instrument blanks are analyzed in the same injection port immediately after any sample for which the target compound exceed the calibration range or when non-target compounds are found at concentrations greater than 100 ug/L or when ions from a compound saturate the detector (excluding peaks in a solvent front).

Blank Acceptance Criteria

Each blank must be analyzed on a GC/MS system that has met criteria for the instrument performance check and initial and continuing calibration.

- The DMC recovery in the blanks must meet the acceptance recovery criteria found in Table 4, Section 18.0. Note the recovery limits for 1,4-dioxane-d_8 are advisory.

- The percent area response change for each of the internal standards in the blank must be within the inclusive range of 50 to 200 percent of its response in the most recent continuing calibration standard analysis.

- The retention time shift for each of the internal standards in the blank must be within ±0.50 minutes of its retention time in the most recent continuing calibration standard analysis.

The concentration of each target compound in the method blank and storage blank must be less than the CRQL except for methylene chloride, acetone, and 2-butanone which must be less than 2 times the CRQL. The concentration of each target compound in the instrument blank must be less than the CRQL.

Corrective Action for Blank Analyses

Any method blank that does not meet criteria must be reanalyzed along with all associated samples.

Any instrument blank that does not meet criteria requires reanalysis of the samples following the instrument blank that have any target compounds detected above the CRQL.

Any storage blank that does not meet criteria must be reanalyzed. If the storage blank contains target compounds above the CRQL, reanalyze associated samples to determine if the contamination occurred during storage or if the problem occurred during the analysis. If upon reanalysis, the storage blank meets criteria, then only the results of reanalysis are reported. If after reanalysis, the storage blank still does not meet criteria, the problem is documented in the case narrative along with corrective or preventive actions taken to prevent recurrence.

9.2 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

MS/MSD are performed when requested by the client. MS/MSD are processed and analyzed in the same manner as field samples and at the same concentration as the most concentrated aliquot from which the parent sample analysis is reported. MS/MSD may not be diluted to get either the spiked or non-spiked analytes within calibration range. Additionally, MS/MSD must not be performed on field, trip or rinsate blank samples.

Acceptance Criteria for MS/MSD Analysis

Each MS/MSD must be analyzed on a GC/MS system that has met criteria for the instrument performance check and initial and continuing calibration.

Each MS/MSD must be analyzed within the contract holding time.

The RT shift for each internal standard in the MS/MSD must be within ±0.50 minutes of the retention times for the internal standards found in the most recent opening CCV

analysis.

The accuracy and prcecison of the MS/MSD should be within the limits given in Table 5, Section 18.0. These control limits are advisory and no further action is required unless it is determined that the frequency of failure is indicative of a systematic problem requiring investigation.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Operation Conditions

Purge Conditions
Configure the purge and trap using the following conditions. Parameters may be optimized as long as the same conditions are used for all standards, samples, and blanks.

Purge Gas: UHP Helium
Purge Flow: ~40 mL/min
Purge Temperature: Ambient (Water)/40°C (Soil)
Purge Time: 11.5 min
Dry Purge Time: 0.5 min
Desorb Preheat Temperature: 250°C
Desorb Temperature: 260°C
Desorb Time 4.0 min
Bake Temperature 270°C
Bake Time 2.5 min
P&T Transfer Line Temperature: 110 °C
P&T Valve Temperature: 110 °C

GC Conditions
The following conditions are recommended and may be optimized as long as the same conditions are used for all standards, samples, and blanks.

Jet Separator	Temperature Program
Injection Port: 150°C	40°C for 4.4 minutes
Jet Separator: 180°C	12.5°C/min to 165
MS Transfer Line: 250°C	30°C/min to 220 for 4.47

Mass Spectrometer Conditions

Electron Energy: 70 eV nominal
Mass Range: 35-300 amu
Ionization Mode: Electron Impact (EI)
Scan Time: >1 scan/sec

10.2 GC/MS Tuning & Ion Abundance

The mass calibration of the system is checked at the beginning of each 12-hour period during which samples or standards are analyzed with a BFB tune standard. The injection time of the acceptable BFB starts the 12-hour analysis window. Within this time period, the analysis of all calibration standards, blanks, and samples must occur. All subsequent standards, samples and QC items associated with a BFB analysis must use the same GC/MS conditions used to acquire the BFB, with the exception of the temperature program. If the closing CCV standard for an analytical sequence meets the acceptance criteria of an opening CCV, then another 12-hour analytical period may begin without the injection of a BFB performance check sample. The new 12-hour analytical period starts at the moment of injection of the passing closing CCV.

To perform the instrument performance check, prepare the 25 ug/mL instrument performance check solution (BFB) and inject 2 uL of BFB standard onto the instrument system to yield an on-column concentration of 50 ug/mL and acquire the data.

The data system acquires the data such that three scans (the peak apex and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required and must be performed using a single scan no more than 20 scans prior to the peak apex. Do not background subtract part of the BFB peak.

Check the results against the ion abundance criteria given in Table 3, Section 18. Criteria must be met prior to further analysis. If criteria are not met, perform necessary corrective actions and repeat the instrument performance check. The BFB must meet all criteria prior to subsequent analysis.

10.3 Initial Calibration

After successful analysis of the instrument performance check standard, calibrate the instrument with a minimum of five concentrations levels. Each calibration level must include all target compounds, DMCs and internal standards.

Prepare the calibration standards using the formulations specified in Appendix A. Allow the standards to warm to room temperature prior to analysis. Load the samples into the autosampler begin the analytical sequence and acquire the data.

The data system calculates the RRF, the percent relative standard deviation (%RSD), and the mean RRF for each target and system-monitoring compound over the working range of the curve. The primary characteristic ions used for quantification are given in Table 1 along with the internal standard assignments. If interference prevents use of the primary quantification ion, the secondary ion given in Table 1 may be used. Because the m- and p-xylene isomers co-elute, a single RRF for m- and p-xylene is calculated using the area response for that peak and the appropriate concentration. Equations for these calculations can be found in Appendix. B.

The following technical acceptance criteria should be met before further analysis. If it is not, investigate the source of the problem, perform instrument maintenance and recalibrate if necessary.

- The RRF at each calibration level for each target compound or system monitoring compound should be greater than or equal to the compound's minimum acceptable response factor given in Table 6.

- The %RSD for each target compound should be less than or equal to the value given in Table 6.

- Up to 2 target compounds and DMCs may fail to meet the minimum RRF criteria and the maximum %RSD criteria and still be acceptable as long as these compounds have a minimum RRF greater than or equal to 0.010 and a %RSD of less than or equal to 40.0%. The exceptions are 1,4 dioxane and 1,4-dioxane-d8 which must have a minimum RRF of greater than or equal to 0.0020 and a %RSD of less than or equal to 50.0%.

10.4 Continuing Calibration Verification (CCV)

Each day prior to analysis, after the instrument performance check standard analyze an opening CCV). An opening CCV is not required in the same 12 hour window as initial calibration. Analyze another CCV (closing CCV) after all samples and blanks have been analyzed, and before the end of the 12-hour analytical window. In this scenario, the 12 hour time-frame is established as the injection time of the instrument performance check standard (BFB). However, in each case where the closing CCV meets the acceptance criteria for an opening CCV, analysis of another instrument performance check standard is not required. In this scenario, the closing CCV serves as the opening CCV and the analytical window is set by the injection of the closing CCV. If the closing CCV does not meet the technical acceptance criteria for an opening CCV, then analysis of a BFB followed by an opening CCV, is required.

Prepare a calibration standard at the mid level concentration (ICAL Level 3) and analyze the standard using the same instrument conditions as used for the initial calibration.

The data system calculates the relative response factors (RRF) for each target and DMC using the primary characteristic ions found in Table 4 and Table 5. The target and DMCs are assigned to the internal standard according to Table 3. For the internal standards, the primary ion is used unless interference is present. If interference is evident, the secondary ion(s) is used. The data system also calculates the %D between the continuing calibration relative response factor and the most recent initial calibration mean relative response factor for each target and DMC. The equations for the above calculations can be found in Appendix B.

The CCV must meet the following criteria before further analysis. If criteria are not met, investigate the source of the problem, perform instrument maintenance and recalibrate if necessary.

- The RRF for each target compound and DMC in the opening CCV must be greater than or equal to the compound's minimum acceptable response factor given in Table 6. For closing CCVs the RRF for each compound must be at least 0.010 (except for 1,4 dioxane and 1,4 dioxane-d_8, which must be at least 0.0020).

- For the opening CCV the percent difference for each target compound and DMC listed in Table 6 should be less than or equal to the value given. For the closing CCV, the percent difference of the RRF must be an inclusive ±50%.

- In the opening CCV up to 2 compounds may fail the RRF and %RSD criteria listed above, but these compounds must have a minimum RRF greater than or equal to 0.010 and the %D must be less than or equal to ±40%. The exceptions are 1,4-dioxane and 1,4-dioxane-d8 which must have a minimum RRF of greater than or equal to 0.0020 and a %D of less than or equal to 50.0%.

- Excluding ions present in the solvent front (carbon dioxide), no quantification ion may saturate the detector.

- If the opening CCV criteria cannot be met system re-calibration is required.

- If the closing CCV criteria is not met, all samples and blanks acquired within that 12-hour analytical window require reanalysis.

11.0 PROCEDURE

11.1 Screen Analysis

Samples should be screened prior to analysis following the procedures given in SOP LM-MV-3810 *VOA Screen Analysis*. The laboratory may choose not to perform screen analysis for samples from which site history is known or available. The results of screen analysis may be used to determine if the concentration of any target compound in a water sample may exceed the calibration range or excessive sample matrix interference may hinder quantification. Despite the screen result, the CLP SOW requires that an original undiluted analysis must be made and reported for all samples. When the laboratory suspects or has evidence of a high concentration sample for target or non-target compounds, for CLP contract work, the laboratory must contact the project administrator (SMO) for further instruction.

11.2 pH determination (Water Samples)

Measure and record the pH for each water sample. If the pH measurement of the sample is > 2, notify appropriate Project Manager. Do not adjust the pH of any sample.

11.3 Sample Preparation

Low Level Water/Soils & Sediment
Allow the samples to warm to ambient temperature. Using a gas tight syringe add a sufficient amount of the internal standard (ISTD) and DMC standards (SSTD) through the septa cap of each vial. Add the appropriate spike volume to each MS/MSD. See the sample preparation table in Appendix A for the volume of standard solution to add to each sample type.

To prepare the method blank and instrument blanks, fill a 44 mL vial with VOA free water and add a sufficient amount of the internal standard (ISTD) and DMC standards (SSTD) through the septa cap of each vial. See the sample preparaiton in Appendix A for spike solution volumes.

To prepare the storage blank, allow the blank to warm to ambient temperature. Using a gas tight syringe add a sufficient amount of the internal standard (ISTD) and DMC standards (SSTD) through the septa cap of each vial. See sample preparation table in Appendix A for the volume of standard solution to add to each sample type

Medium Level Soil/Sediment

Allow the samples to warm to ambient temperature. Using a (INSERT DEVICE) withdraw an 880 μL aliquot of sample and transfer to a 44 mL VOA vial filled with VOA-free reagent water. Using a gas tight syringe add a sufficient amount of the internal standard (ISTD) and DMC standards (SSTD) through the septa cap of each vial. Add the appropriate spike volume to each MS/MSD. See the sample preparation table in Appendix A for the volume of standard solution to add to each sample type.

Sample Analysis

Place samples in autosampler. Create the data acquisition sequence table on the data system, include the necessary sample information and initiate the data collection sequence. Initiate the autosampler sequence. Instrument operations follow the purge and GC/MS conditions outlined in Section 10.1.

11.4 Target Compound Identification

Acquire and process the data. The data system identifies target compounds using their mass spectra and comparing the sample mass spectrum to the mass spectrum of the standard of the suspected compound. The elution of the sample component must be at the same GC relative retention time as the standard component and there must be correspondence of the sample component and standard component mass spectra.

For correspondence, the sample component RRT should compare within ±0.06 RRT of the RRT of the CCV or the mid-point of the initial calibration that was analyzed within the same 12-hour window as the sample. If interference prohibits assignment, assign the RRT by using extracted ion current profiles for the ions unique to the target compound. For comparison of mass spectra, use the mass spectra obtained for the daily calibration standard for identification purposes provided that the instrument met the acceptance criteria for the BFB.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must to be present in the sample spectrum.

- The relative intensities of these ions must agree within ±20% between the standard and sample spectra.

- Ions greater than 10% in the sample spectrum but not present in the standard spectrum are considered and accounted for by the analyst making the comparison.

The data system determines the concentration of target compounds by the internal standard method from the relative response factor (RRF) from the continuing calibration standard using the equations given in Appendix B.

Evaluate the internal standard response and retention time in all samples by comparing the sample internal standard response and retention times to the internal standard response and retention time of the opening continuing calibration standard (or mid-point of calibration curve when samples are analyzed in the same analytical window as the initial calibration). Evaluate the Extracted Ion Current Profile (EICP) of the internal standards for each sample and QC item.

The following criteria should be met for each sample analysis:

- The sample must be acquired on an instrumentation that has met all of the GC/MS tune, calibration, and blank acceptance criteria as defined in Sections 9 and 10.

- All samples and subsequent required dilutions must be analyzed within the contract holding time.

- The area response of the internal standards of the sample must be within 50% to 200% of the response of the internal standards in the most recent opening CCV.

- The retention time shift for each of the internal standard should be within ±0.50 minutes between the sample and the most recent opening CCV.

- The percent recovery for each DMC must be within the acceptance limits found in Table 4. The recovery limits for 1,4-Dioxane-d_8 are advisory. Up to three DMCs (excluding 1,4-Dioxane-d_8) may fail to meet the acceptance criteria listed in Table 4.

- Excluding ions in the solvent front (carbon dioxide), no ion may saturate the detector.

- It must be demonstrated that the sample analyses are free of carryover from a previously analyzed contaminated sample whose target compound response exceeds the calibrated range.

Secondary ion quantification may only be performed when there are sample matrix related interferences with the primary ion. Calculate the RRF using the area response from the most intense secondary ion free of interferences. Document the reason in the project narrative.

If any of the system monitoring or internal standard compounds fail to meet RT or recovery acceptance criteria, investigate for error. If instrument or calculations errors

are found, correct the problem and re-analyze and/or recalibrate as necessary. If it is suspected that the problem is due to matrix effect, re-analyze the sample. If the results of re-analysis are within control, submit only that analysis. Otherwise provide both sets of data using R1/RE designations for the re-analysis. A failing sample with an associated MS/MSD pair is considered confirmed if failures are consistent in all three analyses.

In those instances were the automated processing system fails to accurately integrate quantification ion EICPs due to matrix interference, co-elution, and/or baseline noise, the analyst must perform a manual integration. The manual integration must be made such that it accurately defines the peak without under or over estimation of the absolute area attributable to that component peak. Analysts are to identify all manual integrations by dating and initialing the quantification reports and providing the EICP printout that displays the compound peak before and after manual integration. Generate the manual integration report identifying all samples with manual integrations and note in the case narrative. For additional guidance regarding Manual Integration, refer to laboratory SOP LP-LB-0006 *Manual Integration*.

11.5 Non-Target Compound Identification

Identification of non-target compounds or TICs is not possible using the SIM analysis. SIM data acquisition precludes the collection of mass information necessary for the spectral identification of non-target analytes.

For Trace analysis, perform a library search using the NIST spectral library to tentatively identify up to 30 non-target, non-alkane organic compounds of greatest apparent concentration excluding the system monitoring and internal standards compounds. Assign tentative identification based on mass spectral interpretation.

Do not report the following:

- Substances with responses less than 10 percent of the internal standard.
- Substances that elute earlier than 30 seconds before the first purgeable compound or three minutes after the last purgeable compound.
- Carbon dioxide.
- Semivolatile compounds

TIC peaks that are suspected to be straight chain, branched, or cyclic alkanes must be searched and documentation of the search supplied in the data report. Alkanes are not considered as part of the 30 compound search described earlier. Straight-chain, branched or cyclic alkanes are automatically tabulated and reported as ‘total alkanes” on the Form 1 VOA-TIC. When these alkanes are tentatively identified, the concentrations are considered estimated and reported in the project narrative as alkanes by class.

Determine the estimated concentrations for non-target compounds by the internal

standard method using the same equations specified for target compounds using an assumed RRF of 1. Qualify the resulting concentrations with a "J" and "N" data flag to indicate the concentration is estimated and that there are qualitative and quantitative uncertainties associated with the result.

The following guidelines should be used for making tentative identification:

- If the library search quality match is 85% or better the peak should be reported using the identification generated by the search routine, unless the mass spectral interpretation specialist feels there is just evidence not to report that identification.

- If the library produces more than one match at or above 85%, report the highest match, or first match if they are the same, unless the mass spectral interpretation specialist feels there is just evidence not to report that identification.

- If the library produces multiple matches at or above 85% (e.g. multiple isomers of a PCB), report the highest match, or first match if they are the same, unless the mass spectral interpretation specialist feels there is just evidence not to report that identification.

- If the mass spectral interpretation specialist has just evidence to support reporting a compound with a tentative identification other than the library selection (with 85% quality match or better) detail the justification in the SDG case narrative.

- If the library search produces no matches at or above 85% quality, the mass spectral interpretation specialist should attempt (where possible) to make a tentative identification of the compound, or its class (e.g. alcohol, chlorinated, etc). Otherwise, the compound should be reported as an "Unknown".

12.0 CALCULATIONS

See Appendix B.

13.0 DATA ASSESSMENT, CRITERIA AND CORRECTIVE ACTION

13.1 Data Review and Corrective Action

Review all samples, standards, and QC samples against the acceptance criteria provided in this SOP. If the results do not fall within the criteria, perform corrective action. If corrective action is not taken, or is unsuccessful, document the situation with a nonconformance report and qualify the data using an appropriate data qualifier. For additional guidance regarding the laboratory's protocol and required elements for each level of data review (primary, secondary, and tertiary) refer to laboratory SOP LP-QA-003 *Data Review*.

14.0 METHOD PERFORMANCE

A Method Detection Limit (MDL) must be determined on each instrument used for

analysis for each matrix prior to initial use of the method. The MDLs are then verified annually with an MDL Verification Check sample that is subject to the same extraction and cleanup procedures as used for field samples. Some regulatory programs may require that the MDL study be repeated annually, but this is not a requirement of the CLP SOW. The procedure and acceptance criteria for MDL studies and MDL verficiation are given in laboratory SOP LP-QA-005 *Method Detection Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.1 Waste Streams Produced by the Method

All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide the pollution prevention and waste management procedure described in this SOP and the policies for "Waste Management and Pollution Prevention" given in Section 13 of the Corporate Safety Manual.

The following waste streams are produced from this procedure:

1) Acid Preserved Water Samples & Glass Vials
   → *Satellite Waste Container: 5 Gallon Plastic Bucket labeled "CORROSIVE"*

Transfer the waste stream(s) to the appropriate satellite waste container(s) located in your work area. Notify the Environmental Health & Safety (EHS) Coordinator when the satellite container is near full. The EHS Coordinator will make arrangements for authorized personnel to transfer the waste to the waste management room for subsequent disposal. See laboratory SOP LP-LB-001 *Hazardous Waste* for additional information regarding waste management and disposal procedures.

16.0 REVISION HISTORY

16.1 Title Page: Updated approval signatures.

16.2 Section 2.0: Listed laboratory modifications from reference test method.

17.0 REFERENCES

17.1 Exhibit D, Trace Volatiles, CLP SOW SOM01.0 Multi-Media, Multi-Concentration Statement of Work for Organics Analysis, October 2004.

17.2 CLP SOW Summary of Changes SOM01.0 (10/08/04) to SOM01.1 (05/26/05).

18.0 TABLES, DIAGRAMS & FLOWCHARTS

18.1 Table 1: Target Compound List, Internal Standard Group Assignments, CRQLs, and Quantification and Qualifier Masses
18.2 Table 2: Primary Materials Used
18.3 Table 3: BFB Criteria
18.4 Table 4: DMC Quantification and Qualifier Masses, Internal Standard Group Assignments, and Recovery Limits
18.5 Table 5: Matrix Spike Recovery, Accuracy, and Precision Limits (Advisory)
18.6 Table 6: Calibration Criteria
18.7 Appendix A: Standard Preparation Tables
18.8 Appendix B: Equations
18.9 Appendix C: Terms and Definitions

Table 1: Target Compound List, Internal Standard Group Assignments, CRQLs, and Quantification and Qualifier Masses

Compound	ISTD Group*	Low Water ug/L	Low Soil ug/Kg	Med Soil ug/Kg	MZ1**	MZ2	MZ3
Dichlorodifluoromethane	1	5	5	250	85	87	
Chloromethane	1	5	5	250	50	52	
Vinyl chloride	1	5	5	250	62	64	
Bromomethane	1	5	5	250	94	96	
Chloroethane	1	5	5	250	64	66	
Trichlorofluoromethane	1	5	5	250	101	103	
1,1-Dichloroethene	1	5	5	250	96	61	63
1,1,2-Trichloro-1,2,2-trifluoroethane	1	5	5	250	101	85	151
Acetone	1	10	10	500	43	58	
Carbon disulfide	1	5	5	250	76	78	
Methyl acetate	1	5	5	250	43	74	
Methylene chloride	1	5	5	250	84	49	86
trans-1,2-Dichloroethene	1	5	5	250	96	61	98
Methyl-t-butyl ether	1	5	5	250	73	57	
1,1-Dichloroethane	1	5	5	250	63	65	83
cis-1,2-Dichloroethene	1	5	5	250	96	61	98
2-Butanone	1	10	10	500	43	72	
Bromochloromethane	1	5	5	250	128	49	130
Chloroform	1	5	5	250	83	85	
1,1,1-Trichloroethane	2	5	5	250	97	99	61
Carbon tetrachloride	2	5	5	250	117	119	
Cyclohexane	2	5	5	250	56	69	84
Benzene	2	5	5	250	78		
1,2-Dichloroethane	1	5	5	250	62	98	
1,4-Dioxane	1	100	100	5000	88	43	58
Trichloroethene	2	5	5	250	95	130	132
Methylcyclohexane	2	5	5	250	83	55	98
1,2-Dichloropropane	2	5	5	250	63	112	65
Bromodichloromethane	2	5	5	250	83	85	127
cis-1,3-Dichloropropene	2	5	5	250	75	77	
4-Methyl-2-pentanone	2	10	10	500	43	58	100
Toluene	2	5	5	250	91	92	
trans-1,3-Dichloropropene	2	5	5	250	75	77	
1,1,2-Trichloroethane	2	5	5	250	97	83	85
Tetrachloroethene	2	5	5	250	164	166	129
2-Hexanone	2	5	5	250	43	58	57
Dibromochloromethane	2	5	5	250	129	127	
1,2-Dibromoethane	2	5	5	250	107	109	188

Compound	ISTD Group*	Low Water ug/L	Low Soil ug/Kg	Med Soil ug/Kg	MZ1**	MZ2	MZ3
Chlorobenzene	2	5	5	250	112	114	77
Ethylbenzene	2	5	5	250	91	106	
m,p-Xylene	2	5	5	250	106	91	
o-Xylene	2	5	5	250	106	91	
Styrene	2	5	5	250	104	78	103
Bromoform	3	5	5	250	173	175	254
Isopropylbenzene	2	5	5	250	105	120	77
1,1,2,2-Tetrachloroethane	2	5	5	250	83	85	131
1,3-Dichlorobenzene	3	5	5	250	146	111	148
1,4-Dichlorobenzene	3	5	5	250	146	111	148
1,2-Dichlorobenzene	3	5	5	250	146	111	148
1,2-Dibromo-3-chloropropane	3	5	5	250	75	155	157
1,2,4-Trichlorobenzene	3	5	5	250	180	182	145
1,2,3-Trichlorobenzene	3	5	5	250	180	182	145

*ISTD 1 – 1,4-Difluorobenzene
ISTD 2 – Chlorobenzene-d_5
ISTD 3 – 1,4-Dichlorobenzene-d_4

**Denotes quantification mass

Table 2: Primary Material Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Sodium bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure may cause burn if not flushed with water. May cause mild irritation to skin. Prolonged exposure may cause burn if not flushed with water.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 3: BFB Criteria

Mass	Ion Abundance Criteria
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95*
173	less than 2.0% of mass 174
174	50.0-120.0% of mass 95
175	5.0-9.0% of mass 174
176	95.0-101% of mass 174
177	5.0-9.0% of mass 176

***-** All ion abundances must be normalized to m/z 95, the nominal bas peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95

Table 4: Surrogate: Internal Standard Group Assignment and Recovery and Accuracy Limits

DMC	MZ1	MZ2	MZ3	ISTD Group *	Water		Soil	
					Lower	Upper	Lower	Upper
Vinyl chloride-d3	65	67		1	65	131	68	122
Chloroethane-d5	69	71	51	1	71	131	61	130
1,1-Dichloroethene-d2	63	98	65	1	55	104	45	132
2-Butanone-d5	46	71		1	49	155	20	182
Chloroform-d	84	86	47	1	78	121	72	123
Benzene-d6	84	82		2	77	124	80	121
1,2-Dichloroethane-d4	65	67	51	1	78	129	79	122
1,4-Dioxane-d8**	96	51	66	1	50	150	50	150
1,2-Dichloropropane-d6	67	65	46	2	79	124	74	124
Toluene-d8	98	100	42	2	77	121	78	121
trans-1,3-Dichloropropene-d4	79	81	42	2	73	121	72	130
2-Hexanone-d5	63	46		2	28	135	17	184
1,1,2,2-Tetrachloroethane-d2	84	86		2	73	125	56	161
1,2-Dichlorobenzene-d4	152	150		3	80	131	70	131

*ISTD 1 – 1,4-Difluorobenzene
ISTD 2 – Chlorobenzene-d_5

ISTD 3 – 1,4-Dichlorobenzene-d_4
** Limits for 1,4-Dioxane-d_8 are advisory only

Table 5: Matrix Spike Recovery, Accuracy and Precision Limits (Advisory)

	Water			Soil		
Compound	**Lower**	**Upper**	**RPD**	**Lower**	**Upper**	**RPD**
1,1-Dichloroethene	61	145	0-14	59	172	0-22
Benzene	76	127	0-11	66	142	0-21
Trichloroethene	71	120	0-14	62	137	0-24
Toluene	76	125	0-13	59	139	0-21
Chlorobenzene	75	130	0-13	60	133	0-21

Table 6: Calibration Criteria

Compound	Minimum RRF ICAL & Opening CCV	Maximum %RSD ICAL	Maximum %D Opening CCV	Minimum RRF Closing CCV	Maximum %D Closing CCV
Dichlorodifluoromethane	0.010	40	40	0.010	50
Chloromethane	0.010	40	40	0.010	50
Vinyl chloride	0.100	20	25	0.010	50
Bromomethane	0.100	20	25	0.010	50
Chloroethane	0.010	40	40	0.010	50
Trichlorofluoromethane	0.010	40	40	0.010	50
1,1-Dichloroethene	0.100	20	25	0.010	50
1,1,2-Trichloro-1,2,2-trifluoroethane	0.010	40	40	0.010	50
Acetone	0.010	40	40	0.010	50
Carbon disulfide	0.010	40	40	0.010	50
Methyl acetate	0.010	40	40	0.010	50
Methylene chloride	0.010	40	40	0.010	50
trans-1,2-Dichloroethene	0.010	40	40	0.010	50
Methyl-t-butyl ether	0.010	40	40	0.010	50
1,1-Dichloroethane	0.200	20	25	0.010	50
cis-1,2-Dichloroethene	0.010	40	40	0.010	50
2-Butanone	0.010	40	40	0.010	50
Bromochloromethane	0.050	20	25	0.010	50
Chloroform	0.200	20	25	0.010	50
1,1,1-Trichloroethane	0.100	20	25	0.010	50
Carbon tetrachloride	0.100	20	25	0.010	50
Cyclohexane	0.010	40	40	0.010	50
Benzene	0.400	20	25	0.010	50
1,2-Dichloroethane	0.100	20	25	0.010	50
1,4-Dioxane	0.0020	50	50	0.0020	50
Trichloroethene	0.300	20	25	0.010	50

Compound	Minimum RRF ICAL & Opening CCV	Maximum %RSD ICAL	Maximum %D Opening CCV	Minimum RRF Closing CCV	Maximum %D Closing CCV
Methylcyclohexane	0.010	40	40	0.010	50
1,2-Dichloropropane	0.010	40	40	0.010	50
Bromodichloromethane	0.200	20	25	0.010	50
cis-1,3-Dichloropropene	0.200	20	25	0.010	50
4-Methyl-2-pentanone	0.010	40	40	0.010	50
Toluene	0.400	20	25	0.010	50
trans-1,3-Dichloropropene	0.100	20	25	0.010	50
1,1,2-Trichloroethane	0.100	20	25	0.010	50
Tetrachloroethene	0.100	20	25	0.010	50
2-Hexanone	0.010	40	40	0.010	50
Dibromochloromethane	0.100	20	25	0.010	50
1,2-Dibromoethane	0.010	40	40	0.010	50
Chlorobenzene	0.500	20	25	0.010	50
Ethylbenzene	0.100	20	25	0.010	50
m,p-Xylene	0.300	20	25	0.010	50
o-Xylene	0.300	20	25	0.010	50
Styrene	0.300	20	25	0.010	50
Bromoform	0.050	20	25	0.010	50
Isopropylbenzene	0.010	40	40	0.010	50
1,1,2,2-Tetrachloroethane	0.300	20	25	0.010	50
1,3-Dichlorobenzene	0.600	20	25	0.010	50
1,4-Dichlorobenzene	0.500	20	25	0.010	50
1,2-Dichlorobenzene	0.400	20	25	0.010	50
1,2-Dibromo-3-chloropropane	0.010	40	40	0.010	50
1,2,4-Trichlorobenzene	0.200	20	25	0.010	50
1,2,3-Trichlorobenzene	0.200	20	25	0.010	50
Vinyl Chloride-d3	0.010	20	25	0.010	50
Chloroethane-d5	0.010	40	40	0.010	50
1,1-Dichloroethene-d2	0.010	20	25	0.010	50
2-Butanone-d5	0.010	40	40	0.010	50
Chloroform-d	0.010	20	25	0.010	50
Benzene-d6	0.010	20	25	0.010	50
1,2-Dichloroethane-d4	0.010	20	25	0.010	50
1,4-Dioxane-d8	0.0020	50	50	0.0020	50
1,2-Dichloropropane-d6	0.010	40	40	0.010	50
Toluene-d8	0.010	20	25	0.010	50
trans-1,3-Dichloropropene-d4	0.010	20	25	0.010	50
2-Hexanone-d5	0.010	40	40	0.010	50
1,1,2,2-Tetrachloroethane-d2	0.010	20	25	0.010	50
1,2-Dichlorobenzene-d4	0.010	20	25	0.010	50

Appendix A: Standard Preparation Tables

This appendix provides formulations for the standard solutions used in this procedure and are accurate only when the specified concentrations and volumes are used. Any time the volume or concentration or of any of the components change, the analyst must adjust the preparation accordingly. In general, stock standard solutions of a known purity and/or concentration are purchased from commercial vendors and diluted in an appropriate solvent to the desired concentration. The prepared standard solutions are stored and maintained at the appropriate conditions and are discarded when the expiration date has expired. Expiration dates shall be assigned to the prepared standard solution as specified in Section 7.2 of this SOP. The preparation of all standard solutions must be documented in a logbook designated for that purpose and all standard solutions must be labeled according to the laboratory SOP for standard preparation.

SOM Water and Low/Medium Soil Stock Solutions

CALIBRATION MIX

µg/mL Concentration	Description	Vendor	Cat#	Volume Added µL	Final Conc µg/mL
2000	OLC Mega Mix	Restek	30492	200	100
5000	VOA Ketones	Restek	30006	160	200
2000	Gases	Restek	30042	200	100
1000	Non-Ketone DMC	CamIso	ES-528610X	400	100
5000	Ketone DMC	CamIso	ES-528710X	160	200
NEAT	P&T Grade Methanol			2880	

1,4-DIOXANE CAL MIX

µg/mL Concentration	Description	Vendor	Cat#	Volume Added µL	Final Conc µg/mL
2000	1,4-Dioxane	Restek	30287	1000	1000
12500	1,4-Dioxane-d8	CamIso	ES-528810X	160	1000
NEAT	P&T Grade Methanol			840	

DMC (SSTD)

µg/mL Concentration	Description	Vendor	Cat#	Volume Added uL	Final Conc µg/mL
1000	Non-Ketone DMC	CamIso	ES-528610X	400	100
5000	Ketone DMC	CamIso	ES-528710X	160	200
12500	1,4-Dioxane-d8	CamIso	ES-528810X	800	2500
NEAT	P&T Grade Methanol			2640	

INTERNAL STANDARD (ISTD)

µg/mL Concentration	Description	Vendor	Cat#	Volume Added µL	Final Conc µg/mL
2500	LC VOA ISTD Mix	Restek	30091	160	100
NEAT	P&T Grade Methanol			3840	

MATRIX SPIKE

µg/mL Concentration	Description	Vendor	Cat#	Volume Added µL	Final Conc µg/mL
2500	VOA Matrix Spike Mix	Restek	30005	80	100
NEAT	P&T Grade Methanol			192	

BFB Solution

µg/mL Concentration	Description	Vendor	Cat#	Volume Added µL	Final Conc µg/mL
5000	Tuning Mix	Restek	30003	125	25
NEAT	P&T Grade Methanol			24875	

Calibration, QC, and Sample Preparation Summary

SOM Routine in 44 mL (Water - Prepared in HCl preserved vials)

		Routine				
		SOM ISTD	SOM SSTD	SOM Cal Mix	SOM MS Mix	1,4-Dioxane Cal Mix
	Concentration	100 ppm	50 ppm	100 ppm	50 ppm	1000 ppm
Cal Level 1	**5 ppb**	22 µL		2.2 µL		4.4 µL
Cal Level 2	**10 ppb**	22 µL		4.4 µL		8.8 µL
Cal Level 3 / CCV	**50 ppb**	22 µL		22 µL		55 µL
Cal Level 4	**100 ppb**	22 µL		44 µL		88 µL
Cal Level 5	**200 ppb**	22 µL		88 µL		176 µL
MS / MSD	**50 ppb**	22 µL	22 µL		22 µL	
Samples & Blank		22 µL	22 µL			

SOM Routine in 5 mL (Soil)

		Routine				
		SOM ISTD	SOM SSTD	SOM Cal Mix	SOM MS Mix	1,4-Dioxane Cal Mix
	Concentration	100 ppm	50 ppm	100 ppm	50 ppm	1000 ppm
Cal Level 1	**5 ppb**	Footnote$_1$		Footnote$_1$		Footnote$_1$
Cal Level 2	**10 ppb**	2.5 µL		0.5 µL		1 µL
Cal Level 3 / CCV	**50 ppb**	2.5 µL		1 µL		6.25 µL
Cal Level 4	**100 ppb**	2.5 µL		5 µL		10 µL
Cal Level 5	**200 ppb**	2.5 µL		10 µL		20 µL
MS / MSD	**50 ppb**	2.5 µL	2.5 µL		2.5 µL	
Samples & Blank		2.5 µL	2.5 µL			

1: Prepare Cal Level 1 in 44 mL and transfer 5 mL to a new vial for analysis.

Appendix B: Equations

Relative Response Factor (RRF) Calculation

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

Where:
Ax=Area of quantification mass EICP of compound to be measured
A_{is}= Area of quantification mass EICP of associated internal standard
C_{is}=Concentration of internal standard
C_x=Concentration of compound to be measured

Standard Deviation (SD) Calculation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (X_i - \overline{X})^2}{(n-1)}}$$

Where:
X_i=Each value used to calculate the mean
X= The mean of n values
n=Total number of values

Percent Relative Standard Deviation (%RSD) Calculation

$$\%RSD = \frac{SD_{RRF}}{\overline{X}} X100$$

Where:
SD_{RRF}=Standard Deviation of the initial calibration response factors for each compound.
X= Mean value of the initial calibration response factors for each compound.

Mean Relative Response Factor (RRF) Calculation

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

Where:
X_i=Each RRF value per compound
X= The mean of n values
n=Total number of values

Percent Difference (%D) Calculation

$$\%Difference = \frac{RRF_c - \overline{RRF_i}}{RRF_i} X100$$

Where:
RRF_c=Relative Response Factor from current CCV standard
RRF_i=Mean relative response factor from most recent initial calibration

Water Sample Concentration

$$C_x = \frac{(A_x)(I_s)(DF)}{(A_{is})(\overline{RRF})(V_o)}$$

Where:
C_x = Concentration of compound (μg/L)
I_{is} = Concentration of associated internal standard (μg/L).
DF = Dilution Factor.
A_{is} = Area of quantification ion for associated internal standard.
A_x = Area of quantification ion for compound.
RRF = Relative Response Factor from the initial calibration standard
V_0 = actual volume purged (mL).

Low-Level Soil Concentration

$$C_x = \frac{(A_x)(I_s)(DF)}{(A_{is})(\overline{RRF})(W_s)(D)}$$

Where:
C_x = Concentration of compound (μg/Kg dry weight).
A_x = Area of quantitation ion for compound.
I_{is} = Concentration of associated internal standard (μg/L).
A_{is} = Area of quantification ion for associated internal standard.
RRF = Relative Response Factor from the initial calibration standard.
W_s= Weight of sample added to purge vessel (g).
DF = Dilution Factor.

$$D = \frac{100 - \%Moisture}{100}$$

Medium Level Soil Concentration

$$C_x = \frac{(A_x)(I_s)(AV_t)(1000)(DF)}{(A_{is})(\overline{RRF})(V_a)(W_s)(D)}$$

Where:
C_x = Concentration of compound (μg/Kg dry weight).
A_x = Area of quantification ion for compound.
I_{is} = Concentration of associated internal standard (μg/L).
AV_t=Adjusted extract volume

$AV_t=V_t+(W_s-(W_s \times D))$

(V_t=Initial volume of methanol extract before correction, typically 5.0 or 10.0 mL)
A_{is} = Area of quantification ion for associated internal standard.
RRF = Relative Response Factor from the initial calibration standard.
V_a=sample extract aliquot volume (uL)
W_s= Weight of sample added to purge vessel (g).
DF = Dilution Factor.

$$D = \frac{100 - \%Moisture}{100}$$

DMC Percent Recovery Calculation

$$\%R = \frac{Q_d}{Q_a} X100$$

Where:
Q_d = Concentration or amount determined by analysis.
Q_a = Concentration or amount added to sample/blank.

Matrix Spike/Matrix Spike Duplicate Recovery

$$MatrixSpikeRecovery = \frac{SSR - SR}{SA} x100$$

Where,
SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

Matrix Spike/Matrix Spike Duplicate RPD

$$RPD = \frac{|MSR - MSDR|}{1/2(MSR + MSDR)} x100$$

Where,
MSR = Matrix Spike Recovery
MSDR = Matrix Spike Duplicate Recovery

Appendix C: List of Definitions

BAR GRAPH SPECTRUM: a plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

BLANK - an analytical sample designed to assess specific sources of laboratory contamination.

BFB-compound chose to establish mass spectral instrument performance for volatile analysis.

EXTRACTION ION CURRNE PROFILE (EICP)-a plot of ion abundance versus time (or scan number) for ion(s) of specified mass(es).

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

INTEGRATION SCAN RANGE-the scan number of the scan at the beginnig of the area of integration to the scan number at the end of the area of integration.

INTEGRATION TIME RANGE-the retention time at the beginning of the area of integration to the retention time at the end of the area of integration.

INTERNAL STANDARDS- compound added to every standard, blank, matrix spike, matrix spike duplicate, sample (volatiles) and sample extract (semivolatiles) at a known concentration prior to analysis. Internal standards are used as the basis for quantitation of target compounds.

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards, and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

PERCENT DIFFERENCE (%D) - as used in this SOW and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero.

PRIMARY QUANTITATION ION-a contract specified ion used to quantitate a target analyte.

RECONSTRUCTED ION CHROMATOGRAM (RIC)-a mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

RELATIVE PERCENT DIFFERENCE (RPD) - as used in this SOW and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

RELATIVE RESPONSE FACTOR (RRF)-a measure of the relative mass spectral response of an analyte compared to its internal standard.

RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

RESPONSE - or Instrumental Response: a measurement of the output of the GC detector (MS, EC, or FID) in which the intensity of the signal is proportionate to the amount (or concentration) detected. Measured by peak area or peak height.

RETENTION TIME (RT) - the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SURROGATES (Surrogate Standard) - added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - the date on which a sample is received at the Contractor's

STANDARD OPERATING PROCEDURE
STL BURLINGTON

SOM01.1 ANALYTICAL METHOD FOR SEMIVOLATILES
EPA CLP SOM01.1

Applicable Matrix: Non-Potable Water, Soil & Sediment

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the analysis of water, soil and sediment samples from hazardous waste sites for the determination of the semivolatile organic compounds listed in Table 1.0, Section 18.0 of this document. This procedure is specific to the USEPA Contract Laboratory Program (CLP) Statement of Work SOM01.1.

1.2 The EPA has documented the following problems associated with the following compounds:

- Dichlorobenzidine and 4-chloroaniline can be subject to oxidative losses during solvent concentration.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reactions in acetone solution, and photochemical decomposition.
- N-nitrosodiphenylamine decomposes in the gas chromatograph inlet forming diphenylamine and, consequently, may be detected as diphenylamine.

2.0 SUMMARY OF METHOD

2.1 Samples are extracted, concentrated and subject to cleanup following the procedures given in laboratory SOP LM-OP-SOM01.1-SVOA.

2.2 An aliquot of pre GPC extract is analyzed by GC/FID to evaluate sample concentrations in extract, to determine low level or medium level concentrations. This screen information is provided back to the extraction lab if any medium level extractions are required. In addition, this screen information is used to determine the correct dilutions for the low level analysis.

Extracts are analyzed by GC/MS using the procedure described in this SOP. When requested sample extracts are analyzed for a select group of compounds, including Polynucleur Aromatic Hydrocarbons (PAHs) and pentachlorophenol by GC/MS, using the Selected Ion Monitoring (SIM) technique. Samples are first analyzed by the full scan method and if all polynuclear aromatic hydrocarbons and pentachlorophenol are detected during the full scan analysis using the low-level method, then a SIM analysis is not performed.

2.3 This procedure is based on CLP SOW SOM01.1, Exhibit D Semivolatiles USEPA Contract Laboratory Program Statement of Work for Organic Analyses, Multi-Media, Multi-Concentration, May 2005.

The laboratory has implemented the following modifications to the SOW:

Due to poor or unattainable resolution of mass chromatographic peaks of Phenanthrene-d10 and Anthracene-d10 in the optional SIM mode, the laboratory reassigned the target

compounds associated with Phenanthrene-d10 in the SOW to an internal standard closer to its RT as follows:

Target Compound	Lab Assigned Internal Standard
Pentachlorophenol	Acenaphthene-d8
Phenanthrene	Acenaphthene
Anthracene	Acenaphthene
Fluoranthene	Chrysene-d12

3.0 DEFINITIONS

3.1 See Appendix C for a complete list of definitions.

4.0 INTERFERENCES

4.1 Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines. All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

When using the SIM technique, carryover contamination from the injection port is especially problematic due to increased sensitivity, and routine injection port, guard column and chromatographic column maintenance should be performed to ensure that the analytical system is free of any potential sources of contamination. The late eluting PAH's are particularly susceptible to carryover contamination.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 Primary Materials Used

Table 2, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated

with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in Section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 GC/MS System

Gas Chromatograph: Hewlett-Packard 5890 Series II equipped with CTC, HP7673 injector, Hp7683B injector or equivalent autosampler injection port for split/splitless analysis and all the required accessories, including syringes, septa, injector port liners, and column.

Mass Spectrometer: Hewlett-Packard 5971 MSD, 5972 MSD, 5973MSD, 5975MSD capable of operating in full scan and SIM mode.

Primary Column: Restek Rxi-5ms 30m x 0.25mm x .25 um, or equivalent

Data System: HP Chemsystem Unix based computer. ThruPut Target 3.5 Data Processing Software.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Acetone, methanol, methylene chloride, iso-octane, 2-propanol, and toluene - pesticide residue analysis grade or equivalent. JT Baker or equivalent.

7.2 Standards

Purchase stock standard solutions from commercial vendors. Prepare intermediate and working standards from stock standard solutions by diluting a known volume of stock standard in a methylene chloride to a specified volume. The formulations for the preparation of intermediate and working standards are provided in Appendix A.

Store standard solutions 4^{o}C ($\pm$ 2^{o}C) in Teflon-lined screw cap amber bottles and unless specified, prepare fresh standards every twelve months. Protect all standards from light and store samples, sample extracts and standards separately.

Deuterated Monitoring Compound Spiking Solution: Prepare a standard spiking solution that contains 2-Methylnaphthalene-d_{10} and Fluoranthrene-d_{10}, at a concentration of 0.4 μg/mL. .

Instrument Performance Check Solution: Prepare a solution of decafluorotriphenyl-phosphine (DFTPP), such that a 2µL injection will contain 50 ng of DFTPP.

Routine Initial and Continuing Calibration Solutions: The following calibration concentration levels are based on a 2ul injection: 10, 20, 40, 80 and 160 ug/L. Use the 20 ug/L calibration standard as the continuing calibration standard.

SIM Initial and Continuing Calibration Solutions: Prepare calibration standards at concentration levels 0.10, 0.20, 0.40, 0.80, and 1.0ng/μL. Prepare a four-point calibration for Pentachlorophenol at 0.20, 0.40, 0.80, and 1.0 ug/L. Use the 0.40ng/μL calibration standard as the continuing calibration level standard and prepare this standard weekly.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 Unless otherwise specified by client or regulatory program, sample extracts are stored at 4°C (±2°C) for 30 days after delivery of the data report. After 30 days, the samples are disposed of in accordance with applicable regulations.

8.2 The analytical holding time is 40 days from start of extraction.

9.0 QUALITY CONTROL

9.1 Method Blank

A method blank is extracted with each extraction batch of 20 or less samples and analyzed on each GC/MS system used to analyze the samples. The percent recovery of the DMCs in the blank must be within the acceptance limits given in Table 9. For low level soils and water samples, the concentration of target compounds in the method blank must be less than the CRQL for each target compound and less than five times the CRQL for bis(2-ethylhexyl) phthalate. For medium-level soils, the method blank must contain less than the CRQL of any single target compound. Also, the analysis must meet the same technical criteria and DMC recovery as specified for samples. If these criteria are not met, corrective action must be taken before further sample analysis.

9.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A MS/MSD should be performed for every 20 field samples of a similar matrix per client specification. The percent recovery and the relative percent difference should be within the advisory limits given in Table 7.

9.3 Laboratory Control Sample (LCS)

Although the CLP SOW does not include the LCS as a QC item, an LCS may be performed when requested by the client. In the absence of client specific acceptance limits, the percent recovery of the LCS should be within the advisory limits specified for the MS/MSD.

9.4 Deuterated Monitoring Compound (DMC)

DMCs are added to all samples and the method blank prior to extraction. The percent recovery of the DMC compounds for non-diluted samples must be within the accuracy limits given in Table 9. Up to 4 DMCs per sample may fail to meet the recovery limits listed in Table 9 but all percent recoveries must be greater than zero. If SIM analsyis is performed, both SIM DMCs must be within the recovery limits. If the criteria are not met, corrective action including re-extraction and re-analysis must be taken.

9.5 Internal Standards

Internal standards are added to every standard, blank, matrix spike, matrix spike duplicate, and sample extract prior to analysis. Internal standards are used as the basis for quantitation of target compounds. Each internal standard must be within the range of 50.0% and 200% of the response of the internal standard in the most recent opening CCV standard analysis.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Operating Conditions

Gas Chromatograph (GC)

The following conditions are recommended. Other conditions may be used provided that all technical criteria are met. Once the GC conditions are established, the same conditions must be used for the analysis of all standards, samples and QC samples.

Initial Column Conditions: 35ºC for 2 minutes.
Column Temperature: 35ºC to 320ºC at 14 /min.
Final Temperature: 320ºC for 5.6 min, or until Benzo(g,h,i)perylene has eluted
Injector Temperature: 225ºC
Transfer Line Temperature: 300ºC
Injector: Grob-like, splitless
Sample volume: 2µL
Carrier Gas: Helium

Mass Spectrometer (MS)

Electron Energy: 70 volts
Mass Range: 35-500 amu
Scantime: Not to exceed 1 second per scan

SIM Operating Conditions
Column Temperature: 45ºC to 320ºC at 12 /min.
Final Temperature: 320ºC until Benzo(g,h,i)perylene has eluted
Injector Temperature: 225ºC

Transfer Line Temperature: 300ºC
Injector: Grob-like, splitless
Sample volume: 2μL
Carrier Gas: Helium

10.2 GC/MS Mass Calibration (Tune) and Ion Abundance Check

Prior to analysis the GC/MS system is tuned to meet manufacturer's specification using an instrument performance check standard (DFTPP). The mass calibration and resolution of the system must be within criteria prior to subsequent analysis. The injection time of the compliant DFTPP establishes the 12 hour analytical window, thus the DFTPP must be analyzed at the beginning of each 12-hour period during which samples or standards are analyzed except when a closing continuing calibration verification (CCV) can be used as an opening CCV for the next 12-hour period, then an additional DFTPP is not required and the 12-hour period begins at the time of injection of the closing CCV standard.

For SIM analysis, the CLP SOW does not require analysis of an instrument performance check sample and the time-frame of the analytical window is established by the injection time of the first calibration standard or the first CCV when calibration is not performed.

To analyze the instrument performance check standard, inject 2uL of the 25ng/μL DFTPP solution to yield an on-column concentration of 50 ng/uL and acquire the data.

The data processing system is set to sum three spectral points (the apex scan, the scan prior to the apex, and the scan immediately following the apex), and to subtract a background scan. Background subtraction is required and must be accomplished using a single scan no more than 20 scans prior to the elution of the DFTPP.

Check to ensure the ion abundance criteria shown in Table 2 is met before analysis of calibration standards. If the criteria are not met, perform corrective action. If the corrective action taken may affect the tune of the instrument, re-verify the tune with another DFTPP analysis prior to further analysis.

10.3 Initial Calibration

After successful analysis of the instrument performance check standard, calibrate the instrument with a minimum of five concentrations levels. Each calibration level must include all target compounds, DMCs and internal standards.

Prepare the calibration standards using the formulations specified in Appendix A. Allow standards to warm to room temperature prior to preparation. Inject 2uL of each calibration standard to which internal standards are added to result a concentration of 20ng/uL onto the GC/MS system and acquire the data.

The data system calculates the relative response factors (RRF) for each target and DMC. The target compounds and DMCs are assigned to the internal standard according to Table 3. For the internal standards, the primary ion is used unless interference is present. If interference is evident, the secondary ion(s) is used. The data system also calculates the mean relative response factor for all compounds and the % relative

standard deviation (%RSD) of the RRF values for the initial calibration. Equations for the above calculations can be found in Appendix B.

The following technical acceptance criteria should be met before further analysis. If it is not, investigate the source of the problem, perform instrument maintenance and recalibrate if necessary.

- The RRF at each calibration concentration for each compound and DMC should be greater than or equal to the compounds minimum acceptable RRF listed in Table 6.

- The %RSD over the initial calibration range for the RRF for each compound and DMC that has a required %RSD should be less than or equal to the %RSD listed in Table 6.

  Note: Up to four compounds may fail to meet criteria listed in Table 6. However, these 4 compounds must meet a minimum RRF criterion of 0.010 and have a %RSD less than or equal to 40 percent.

- Excluding ions with a solvent front, no quantification ion may saturate the detector.

If criteria are met and if time remains in the 12-hour time period samples may be analyzed without continuing calibration verification (CCV).

Repeat initial calibration when corrective actions that affect the calibration criteria has occurred (e.g. ion source cleaning, repair, column replacement) or when continuing calibration acceptance criteria have not been met.

10.4 Continuing Calibration Verification (CCV)

Each day prior to analysis, after the instrument performance check standard analyze a continuing calibration verification standard (opening CCV). An opening CCV is not required in the same 12 hour window as initial calibration. Analyze another CCV (closing CCV) after all samples and blanks have been analyzed, and before the end of the 12-hour analytical window. In this scenario, the 12 hour time-frame is established as the injection time of the instrument performance check standard (DFTPP). However, in each case where the closing CCV meets the acceptance criteria for an opening CCV, analysis of another instrument performance check standard is not required. In this scenario, the closing CCV serves as the opening CCV and the analytical window is set by the injection of the closing CCV. If the closing CCV does not meet the technical acceptance criteria for an opening CCV, then analysis of a DFTPP tune, followed by an opening CCV, is required.

Prepare a calibration standard at the mid level concentration. Inject 2uL of the continuing calibration standard which also contains internal standards at 20ng/uL and acquire the data

The data system calculates the relative response factors (RRF) for each target and DMC using the primary characteristic ions found in Table 4 and Table 5. The target and DMCs

are assigned to the internal standard according to Table 3. For the internal standards, the primary ion is used unless interference is present. If interference is evident, the secondary ion(s) is used. The data system also calculates the %D between the continuing calibration relative response factor and the most recent initial calibration mean relative response factor for each target and DMC. The equations for the above calculations can be found in Appendix B.

The CCV must meet the criteria before further analysis. If criteria are not met, investigate the source of the problem, perform instrument maintenance and recalibrate if necessary.

- The RRF for each compound and DMC should be greater than or equal to the compounds minimum acceptable RRF listed in Table 6. For the closing CCV, the
- RRF percent difference for each semivolatile target compound and DMC must be greater than or equal to 0.010.

- The RRF percent difference for each target compound and DMC must be within the inclusive range listed in Table 6. For a closing CCV, the RRF percent difference for each compound must be in the inclusive range of ±50.

  Note: Up to four compounds may fail to meet the minimum RRF or maximum percent difference criteria listed in Table 4, but the RRFs of those 4 compounds must be greater than or equal to 0.010 and the percent differences must be ± 40 percent. For a closing CCV, all target compounds must meet the requirementslisted above for technical acceptance of a closing CCV.

- Excluding ions with a solvent front, no quantification ion may saturate the detector.

11.0 PROCEDURE

11.1 Sample Analysis

Add the internal standard solution to an aliquot of each sample extract to result in a 20ng/ul concentration for full-scan or 0.2ng/uL for the optional SIM analysis of each internal standard. Load the autosampler tray, initiate the analytical sequence, and acquire the data.

11.2 Qualitative Identification of Target Compounds

Target compounds are identified by comparing the sample mass spectrum to the reference standard of the suspected compound. The compound is present if the elution of the sample analyte is within the relative retention time window established from the continuing calibration verification standard and if the sample analyte and calibration standard component mass spectra correspond. The RRT of the sample component should compare within ±0.06 RRT units of the RRT standard component. If samples were analyzed immediately following the initial calibration, compare the analyte retention

times to the mid point calibration standard. For target analytes use the spectra generated from the working calibration standard as the primary spectral reference.

The following requirements should be met for qualitative identification:

- 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.

- The relative intensities of these ions should agree within plus or minus 20% between the standard and sample spectra.

- Ions greater than 10% in the sample spectrum but not present in the standard spectrum may be considered and accounted for by the analyst making the comparison. The verification process should favor false positives. When target compounds are found below the CRQL but the spectrum meets the identification criteria, report the concentration with a "J" data qualifier.

- If a compound cannot be verified by all of the criteria but in the technical judgment of the mass spectra interpretation of the analyst, the identification is considered correct, report the concentration.

11.3 Quantitative Identification of Target Compounds

Target compounds are quantified by the internal standard method. The internal standard used is the one assigned to that analyte for quantification as specified in Table 3. The EICP area of the primary characteristic ions of analytes listed in Tables 4 and Table 5 are used for quantification.

Use the relative response factor (RRF) from the continuing calibration analysis to calculate the concentration in the sample using the equations given in Section 12.0. For samples analyzed in the same 12-hour window as the initial calibration standards use the RRF value from the 50ng standard. Employ secondary ion quantification only when there is sample interference from the primary ion. If secondary ion quantification is performed, note the reason in the project narrative.

If the on-column concentration of any compound in any sample exceeds the initial calibration range, that sample extract must be diluted. Add the internal standard solution to the diluted extract for a concentration of 20 ng/uL of each internal standard. Guidance in performing dilution and exceptions to this requirement are given below.

Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range. The dilution factor chosen should keep the response of the largest peak for a target compound in the upper half of the calibration range of the instrument. Do not submit data for more than two analyses, i.e., the original sample extract and one dilution, or if the samples were

screened, from the most concentrated dilution analyzed and one further dilution. The maximum dilution factor permitted for low level soils is 30. If a low level soil requires a dilution factor greater than 30 in order to bring target compounds within calibration range, then the medium level method shall be used.

11.4 Qualitative Identification of Non-Target Compounds

Perform a library search for non-target compounds using the NIST spectral library. Up to 30 organic compounds of greatest apparent concentration, excluding the internal standard compounds, DMCs and volatile and semivolatile target compounds, shall be identified using the library search.

Up to 20 peaks of greatest apparent concentration that are suspected to be straight-chain, branched or cyclic alkanes, alone or part of an alkane series must be library searched and documentation of the search must be provided in the data report. The concentration of each alkane is to be summed and reported as a single result as “total alkanes”. Alkanes are not considered a part of the TIC described in the section above.

Peaks that are suspected to be aldol-condensation reaction products shall be searched, reported and counted as part of the 30 most intense non-target compounds and qualified with an “A” flag on the deliverable form.

Use the following guidelines for making tentative identification:

- Relative intensities of major ions in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be evaluated for possible background contamination or presence of co-eluting compounds.
- Ions present in the sample spectrum but not in the reference spectrum should be evaluated to determine if background contamination or co-eluting compounds are responsible for the discrepancy. Data system library reduction programs can sometimes create these discrepancies.
- If in the analyst’s technical judgment of the mass spectral interpretation no valid tentative identification can be made, the compound should be reported as unknown. Additional classification of the compound should be given if possible (i.e. unknown aromatic). Additionally, if library search produces a match at or above 85%, report that compound. If library search produces more than one compound at or above

85%, report the first compound. If library search produces no matches at or above 85%, report the compound as unknown.

Do not report the following:

- Substances with responses less than 10 percent of the internal standard
- Substances that elute earlier than 30 seconds before the first compound or three minutes after the last target compound listed in Table 1.
- Volatile compounds

11.5 Quantitative Identification of Non-Target Compounds

An estimated concentration for non-target compounds is generated using the internal standard method using the nearest internal standard free of interferences. The equations for calculation of concentration are given in Section 12.0. Total area counts from the total ion chromatograms are used for both the internal standard and the compound to be measured. Assume a relative response factor of (1) and qualify the resulting concentration with a “J” and an “N” data qualifier to indicate qualitative and quantitative uncertainty.

11.6 Acceptance Criteria for Sample Analysis

The following sample acceptance criteria should be met before data are reported. If criteria are not met, check the calculations, sample preparation procedure, standard solutions and instrument operation. If problems are found, re-extract and/or reanalyze as necessary.

- The samples must be analyzed on a GC/MS system meeting the DFTPP, initial, continuing calibration and blank acceptance criteria and the sample must undergo cleanup procedures when required on a GPC that has met acceptance criteria for GPC calibration.

- The samples must have an associated method blank that meets all criteria.

- Samples must have acceptable DMC recovery using the acceptance range in Table 8.

- The EICP area for each of the internal standards must be within the inclusive range of –50% percent and +200 percent of the response of the internal standards in the most recent continuing calibration analysis.

- The retention time shift for each of the internal standards must be within ±0.50 minutes between the sample and the most recent continuing calibration analysis.

- Excluding those ions in the solvent front, no ion may saturate the detector and no target compound may exceed the upper limit of the calibration range unless a more dilute aliquot of the sample extract is also analyzed.

12.0 CALCULATIONS

12.1 See Appendix B.

13.0 DATA ASSESSMENT, CRITERIA AND CORRECTIVE ACTION

13.1 Data Review and Corrective Action

Review all samples, standards, and QC samples against the acceptance criteria provided in this SOP. If the results do not fall within the criteria, perform corrective action. If corrective action is not taken, or is unsuccessful, document the situation with a nonconformance report and qualify the data using an appropriate data qualifier. For additional guidance regarding the laboratory's protocol and required elements for each level of data review (primary, secondary, and tertiary) refer to laboratory SOP LP-QA-003 *Data Review*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) must be determined on each instrument used for analysis for each matrix prior to initial use of the method. The MDLs are then verified annually with an MDL Verification Check sample that is subject to the same extraction and cleanup procedures as used for field samples. Some regulatory programs may require that the MDL study be repeated annually, but this is not a requirement of the CLP SOW. The procedure and acceptance criteria for MDL studies and MDL verficiation are given in laboratory SOP LP-QA-005 *Method Detection Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 Waste Streams Produced by the Method

The following waste streams are produced when this procedure is performed:

Vials containing sample extracts: Satellite Container

Transfer the waste stream(s) to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in the laboratory SOP for Hazardous Waste.

16.0 REVISION HISTORY

16.1 Title Page: Updated to current management

16.2 Section 2.0: Inserted reference to CLP SOW and included a list of modifications from reference test method.

16.3 Section 16.0: Inserted revision history, moved references to section 17.0.

17.0 REFERENCES

17.1 CLP SOW SOM01.0 Multi-Media, Multi-Concentration Statement of Work for Organics Analysis, October 2004.

17.2 CLP SOW Summary of Changes SOM01.0 (10/08/04) to SOM01.1 (05/26/05).

18.0 TABLES, DIAGRAMS & FLOWCHARTS

18.1 Table 1: Target Compound List and Reporting Limits
18.2 Table 2: Primary Materials Used
18.3 Table 3: DFTPP Key Ions and Abundance Criteria
18.4 Table 4: Internal Standards with Corresponding Target Compounds and Surrogates
18.5 Table 5: Characteristic Ions for Internal Standards
18.6 Table 6: Characteristic Ions for Target Compounds and Surrogates.
18.7 Table 7: RRF Criteria
18.8 Table 8: Matrix Spike Recovery and RPD limits.
18.9 Table 9: Deuterated Monitoring Compound Recovery Limits.
18.10 Appendix A: Standard Preparation Tables
18.11 Appendix B: Equations
18.12 Appendix C: Terms and Definitions

Table 1: Target Compound List and CRQL

Compound	Quantitation Limits				
	Water by SIM (ug/L)	Water (ug/L)	Soil by SIM ug/Kg	Low Soil (ug/Kg)	Med. Soil (ug/Kg)
Benzaldehyde		5.0		170	5000
Phenol		5.0		170	5000
Bis(2-chloroethyl) ether		5.0		170	5000
2-Chlorophenol		5.0		170	5000
2-Methylphenol		5.0		170	5000
2,2'-Oxybis(1-chloropropane)		5.0		170	5000
Acetophenone		5.0		170	5000
4-Methylphenol		5.0		170	5000
N-Nitroso-di-n-propylamine		5.0		170	5000
Hexachloroethane		5.0		170	5000
Nitrobenzene		5.0		170	5000
Isophorone		5.0		170	5000
2-Nitrophenol		5.0		170	5000
2,4-Dimethylphenol		5.0		170	5000
Bis(2-chloroethoxy) methane		5.0		170	5000
2,4-Dichlorophenol		5.0		170	5000
Napthalene	0.10	5.0	3.3	170	5000
4-Chloroaniline		5.0		170	5000
Hexachlorobutadiene		5.0		170	5000
Caprolactum		5.0		170	5000
4-Chloro-3-methylphenol		5.0		170	5000
2-Methylnaphthalene	0.10	5.0	3.3	170	5000
Hexachlorocyclopentadiene		5.0		170	5000
2,4,6-Trichlorophenol		5.0		170	5000
2,4,5-Trichlorophenol		5.0		170	5000
1,1'Biphenyl		5.0		170	5000
2-Chloronaphthalene		5.0		170	5000
2-Nitroaniline		10.0		330	10000
Dimethylphthalate		5.0		170	5000
2,6-Dinitrotoluene		5.0		170	5000
Acenaphthylene	0.10	5.0	3.3	170	5000
3-Nitroaniline		10.0		330	10000
Acenaphthene	0.10	5.0	3.3	170	5000
2,4-Dinitrophenol		10.0		330	10000
4-Nitrophenol		10.0		330	10000
Dibenzofuran		5.0		170	5000
2,4-Dinitrotoluene		5.0		170	5000
Diethylphthalate		5.0		170	5000
Fluorene	0.10	5.0	3.3	170	5000
4-Chlorophenyl-phenyl ether		5.0		170	5000

Table 1: Continued

Compound	Quantitation Limits				
	Water by SIM (ug/L)	Low Water (ug/L)	Soil by SIM (ug/Kg)	Low Soil (ug/Kg	Med. Soil (ug/Kg)
4-Nitroaniline		10.0		330	10000
4,6-Dinitro-2-methylphenol		10.0		330	10000
N-Nitrosodiphenylamine		5.0		170	5000
1,2,4,5-Tetrachlorobenzene		5.0		170	5000
4-Bromophenyl-phenyl ether		5.0		170	5000
Hexachlorobenzene		5.0		170	5000
Atrazine		5.0		170	5000
Pentachlorophenol	0.20	10.0	6.7	330	10000
Phenanthrene	0.10	5.0	3.3	170	5000
Anthracene	0.10	5.0	3.3	170	5000
Carbazole		5.0		170	5000
Di-n-butylphthalate		5.0		170	5000
Fluoranthene	0.10	5.0	3.3	170	5000
Pyrene	0.10	5.0	3.3	170	5000
Butylbenzyl phthalate		5.0		170	5000
3,3-Dichlorobenzidine		5.0		170	5000
Benzo(a)anthracene	0.10	5.0	3.3	170	5000
Chrysene	0.10	5.0	3.3	170	5000
Bis(2-ethylhexyl) phthalate		5.0		170	5000
Di-n-octyl-phthalate		5.0		170	5000
Benzo(b)fluoranthene	0.10	5.0	3.3	170	5000
Benzo(k)fluroanthene	0.10	5.0	3.3	170	5000
Benzo(a)pyrene	0.10	5.0	3.3	170	5000
Indeno(1,2,3-cd)pyrene	0.10	5.0	3.3	170	5000
Dibenzo(a,h)anthracene	0.10	5.0	3.3	170	5000
Benzo(g,h,i)perylene	0.10	5.0	3.3	170	5000
2,3,4,6-Tetrachlorophenol		5.0		170	5000

Table 2: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 3: DFTPP Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
51	10.0-80% of mass 198
68	Less than 2.0% of mass 69
69	Present
70	Less than 2.0% of mass 69
127	10.0-80.0% of mass 198
197	Less than 2.0% of mass 198
198	Base peak, 100% relative abundance
199	5.0-9.0% of mass 198
275	10.0-60.0% of mass 198
365	Greater than 1.0% of mass 198
441	Present, but less than mass 443
442	50.0-100% of mass 198
443	15.0-24.0 percent of mass 442

NOTE: All ion abundance MUST be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be up to 100 percent that of m/z 198.

Table 4: Internal Standards with Corresponding Target Compounds and Surrogates

1,4-Dichlorobenze-d4	Naphthalene-d8	Acenaphthene-d10
Benzaldehyde	Nitrobenzene	Hexachlorocyclopentadiene
Phenol	Isophorone	2,4,6-Trichlorophenol
Bis(2-chloroethyl)ether	2-Nitrophenol	2,4,5-Trichlorophenol
2-Chlorophenol	2,4-Dimethylphenol	2,3,4,6-Tetrachlorophenol
2-Methylphenol	Bis(2-chloroethoxy)methane	1,1'Biphenyl
2,2'-Oxybis(1-chloropropane)	2,4-Dichlorophenol	2-Chloronaphthalene
Acetophenone	4-Chloroaniline	2-Nitroaniline
4-Methylphenol	Hexachlorobutadiene	Dimethylphthalate
N-Nitroso-di-n-propylamine	Caprolactum	Acenaphthylene
Hexachloroethane	4-Chloro-3-methylphenol	3-Nitroaniline
Phenol-d5 (DMC)	2-Methylnaphthalene	Acenaphthene
Bis(2-chloroethyl)ether-d8 (DMC)	Naphthalene	2,4-Dinitrophenol
2-Chlorophenol-d4 (DMC)	Nitrobenzene-d5 (DMC)	4-Nitrophenol
4-Methylphenol-d8 (DMC)	2-Nitrophenol-d4 (DMC)	Dibenzofuran
	2,4-Dichlorophenol-d3 (DMC)	2,4-Dinitrotoluene
	4-Chloroaniline-d4 (DMC	2,6-Dinitrotoluene
		1,2,4,5-Tetrachlorobenzene
		Pentachlorophenol (SIM)
		Phenanthrene(SIM)
		Anthracene(SIM)
		Diethylphthalate
		4-Chlorophenyl-phenyl ether
		Fluorene
		4-Nitroaniline
		Acenaphthylene-d8 (DMC)
		4-Nitrophenol-d4 (DMC)
		Dimethylphthalate-d6 (DMC)
		Fluorene-d10 (DMC)

Phenanthrene-d10	Chrysene-d12	Perylene-d12
4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl phthalate
N-Nitrosodiphenylamine	Butylbenzylphthalate	Benzo(b)fluoranthene
4-Bromophenyl-phenylether	3,3'Dichlorobenzidine	Benzo(k)fluroanthene
Hexachlorobenzene	Benzo(a)anthracene	Benzo(a)pyrene
Atrazine	Bis(2-ethylhexyl)phthalate	Indeno(1,2,3-cd) Pyrene
Pentachlorophenol	Chrysene	Dibenzo(a,h) anthracene
Phenanthrene	Pyrene-d10 (DMC)	Benzo(g,h,i)perylene
Anthracene		Benzo(a)pyrene-d12 (DMC)
Carbazole	Fluoranthene (SIM)	
Di-n-butylphthalate		
Fluroanthene		
4,6-Dinitro-2-methylphenol –d2 (DMC)		
Anthracene-d10 (DMC)		

Table 5: Characteristic Ions for Internal Standards

Internal Standards	Primary Quantitation Ion	Secondary Ion(s)
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

Table 6: Characteristic Ions for Target Compounds and Surrogates

Parameter	Primary Quantitation Ion	Secondary Ion(s)
Benzaldehyde	77	105, 106
Phenol	94	65, 66
Bis(2-chloroehyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
2-Methylphenol	108	107
2,2'-Oxybis(1-chloropropane)	45	77, 79
Acetophenone	105	77, 51
4-Methylphenol	108	107
N-nitroso-di-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
Bis(2-chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
Caprolactum	113	55, 56
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
1,1'Biphenyl	154	153, 76
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182

Table 6: Continued

2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
1,2,4,5-Tetrachlorobenzene	216	214, 179, 108, 143, 218
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249
Atrazine	200	173, 215
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Carbazole	167	166, 139
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
Bis(2-ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-Octyl phthalate	149	None
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenzo(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
2,3,4,6-Tetrachlorophenol	232	131, 230, 166, 234, 168
Deuterated Monitoring Compounds		
Phenol-d5	71	42, 99
Bis(2-chloroethyl)ether-d8	67	99, 69
2-Chlorophenol-d4	132	134, 68, 66
4 Methylphenol-d8	113	115, 54
Nitrobenzene-d5	128	82, 54
2-Nitrophenol-d4	143	69, 41, 42
2,4-Dichlorophenol-d3	165	167, 101
4-Chloroaniline-d4	131	133, 69
Dimethylphthalate-d6	166	78
Acenaphthylene-d8	160	80, 158
4-Nitrophenol-d4	143	113, 41, 42
Fluorene-d10	176	174, 87, 86
4,6-Dinitro-2-methylphenol-d2	200	170, 52
Anthracene-d10	188	94, 80
Pyrene-d10	212	106, 104
Benzo(a)pyrene-d12	264	132, 118

Table 7: RRF Criteria

Parameter	Minimum RRF	Maximum %RSD	Maximum %Diff
Benzaldehyde	0.010	40.0	±40.0
Phenol	0.800	20.0	±25.0
Bis(2-chloroehyl)ether	0.700	20.0	±25.0
2-Chlorophenol	0.800	20.0	±25.0
2-Methylphenol	0.700	20.0	±25.0
2,2'-Oxybis(1-chloropropane)	0.010	40.0	±40.0
Acetophenone	0.010	40.0	±40.0
4-Methylphenol	0.600	20.0	±25.0
N-nitroso-di-n-propylamine	0.500	20.0	±25.0
Hexachloroethane	0.300	20.0	±25.0
Nitrobenzene	0.200	20.0	±25.0
Isophorone	0.400	20.0	±25.0
2-Nitrophenol	0.100	20.0	±25.0
2,4-Dimethylphenol	0.200	20.0	±25.0
Bis(2-chloroethoxy)methane	0.300	20.0	±25.0
2,4-Dichlorophenol	0.200	20.0	±25.0
Naphthalene	0.700	20.0	±25.0
4-Chloroaniline	0.010	40.0	±40.0
Hexachlorobutadiene	0.010	40.0	±40.0
Caprolactum	0.010	40.0	±40.0
4-Chloro-3-methylphenol	0.200	20.0	±25.0
2-Methylnaphthalene	0.400	20.0	±25.0
Hexachlorocyclopentadiene	0.010	40.0	±40.0
2,4,6-Trichlorophenol	0.200	20.0	±25.0
2,4,5-Trichlorophenol	0.200	20.0	±25.0
1,1'Biphenyl	0.010	40.0	±40.0
2-Chloronaphthalene	0.800	20.0	±25.0
2-Nitroaniline	0.010	40.0	±40.0
Dimethylphthalate	0.010	40.0	±40.0
2,6-Dinitrotoluene	0.200	20.0	±25.0
Acenaphthylene	0.900	20.0	±25.0
3-Nitroaniline	0.010	40.0	±40.0
Acenaphthene	0.900	20.0	±25.0
2,4-Dinitrophenol	0.010	40.0	±40.0
4-Nitrophenol	0.010	40.0	±40.0
Dibenzofuran	0.800	20.0	±25.0
2,4-Dinitrotoluene	0.200	20.0	±25.0
Diethylphthalate	0.010	40.0	±40.0
1,2,4,5-Tetrachlorobenzene	0.010	40.0	±40.0
4-Chlorophenyl-phenylether	0.400	20.0	±25.0
Fluorene	0.900	20.0	±40.0
4-Nitroaniline	0.010	40.0	±40.0
4,6-Dinitro-2-methylphenol	0.010	40.0	±40.0
4-Bromophenyl-phenylether	0.100	20.0	±25.0
N-Nitrosodiphenylamine	0.010	40.0	±40.0
Hexachlorobenzene	0.100	20.0	±25.0

Table 7: Continued

Atrazine	0.010	40.0	±40.0
Pentachlorophenol	0.050	20.0	±25.0
Phenanthrene	0.700	20.0	±25.0
Anthracene	0.700	20.0	±25.0
Carbazole	0.010	40.0	±40.0
Di-n-butylphthalate	0.010	40.0	±40.0
Fluoranthene	0.600	20.0	±25.0
Pyrene	0.600	20.0	±25.0
Butylbenzylphthalate	0.010	40.0	±40.0
3,3'-Dichlorobenzidine	0.010	40.0	±40.0
Benzo(a)anthracene	0.800	20.0	±25.0
Chrysene	0.700	20.0	±25.0
Bis(2-ethylhexyl) phthalate	0.010	40.0	±40.0
Di-n-octylphthalate	0.010	40.0	±40.0
Benzo(b)fluoranthene	0.700	20.0	±25.0
Benzo(k)fluoranthene	0.700	20.0	±25.0
Benzo(a)pyrene	0.700	20.0	±25.0
Indeno(1,2,3-cd)pyrene	0.500	20.0	±25.0
Dibenzo(a,h)anthracene	0.400	20.0	±25.0
Benzo(g,h,I)perylene	0.500	20.0	±25.0
2,3,4,6-Tetrachlorophenol	0.100	20.0	±25.0
Deuterated Monitoring Compounds			
Phenol-d5	0.010	20.0	±25.0
Bis(2-chloroethyl)ether-d8	0.010	20.0	±25.0
2-Chlorophenol-d4	0.010	20.0	±25.0
4 Methylphenol-d8	0.010	20.0	±25.0
Nitrobenzene-d5	0.010	20.0	±25.0
2-Nitrophenol-d4	0.010	20.0	±25.0
2,4-Dichlorophenol-d3	0.010	20.0	±25.0
4-Chloroaniline-d4	0.010	40.0	±40.0
Dimethylphthalate-d6	0.010	40.0	±40.0
Acenaphthylene-d8	0.010	20.0	±25.0
4-Nitrophenol-d4	0.010	40.0	±40.0
Fluorene-d10	0.010	20.0	±25.0
4,6-Dinitro-2-methylphenol-d2	0.010	40.0	±40.0
Anthracene-d10	0.010	20.0	±25.0
Pyrene-d10	0.010	20.0	±25.0
Benzo(a)pyrene-d12	0.010	20.0	±25.0

Table 8: Matrix Spike Recovery and Relative Percent Difference Limits

Compound	Water		Soil	
	%R	%RPD	%R	%RPD
Phenol	12-110	0-42	26-90	0-35
2-Chlorophenol	27-123	0-40	25-102	0-50
N-Nitroso-di-n-propylamine	41-116	0-38	41-126	0-38
4-Chloro-3-Methylphenol	23-97	0-42	26-103	0-33
Acenaphthene	46-118	0-31	31-137	0-19
4-Nitrophenol	10-80	0-50	11-114	0-50
2,4-Dinitrotoluene	24-96	0-38	28-89	0-47
Pentachlorophenol	9-103	0-50	17-109	0-47
Pyrene	26-127	0-31	35-142	0-36

Table 9: Deuterated Monitoring Compound Recovery Limits

Compound	Water	Soil
	%R	%R
Phenol-d5	39-106	17-103
Bis(2-chloroethyl) ether-d8	40-105	12-98
2-Chlorophenol-d4	41-106	13-101
4 Methylphenol-d8	25-111	8-100
Nitrobenzene-d5	43-108	16-103
2-Nitrophenol-d4	40-108	16-104
2,4-Dichlorophenol-d3	37-105	23-104
4-Chloroaniline-d4	1-145	1-145
Dimethylphthalate-d6	47-114	43-111
Acenaphthylene-d8	41-107	20-97
4-Nitrophenol-d4	33-116	16-166
Fluorene-d10	42-111	40-108
4,6-Dinitro-2-methylphenol-d2	22-104	1-121
Anthracene-d10	44-110	22-98
Pyrene-d10	52-119	51-120
Benzo(a)pyrene-d12	32-121	43-111

Appendix A: Standard Preparation Tables

This appendix provides formulation tables for the standard solutions used in this procedure and are accurate only when the specified concentrations and volumes are used. Any time the volume or concentration or of any of the components change, the analyst must adjust the preparation accordingly. In general, stock standard solutions of a known purity and/or concentration are purchased from commercial vendors and diluted in an appropriate solvent to the desired concentration. The prepared standard solutions are stored and maintained at the appropriate conditions and are discarded when the expiration date has expired. Expiration dates shall be assigned to the prepared standard solution as specified in Section 7.2 of this SOP. The preparation of all standard solutions must be documented in a logbook designated for that purpose and all standard solutions must be labeled according to the general laboratory SOP for standard preparation.

SOM01.1 Calibration Mix

Component	Vendor	Catalog #	Concentration (ug/mL)	Volume Added (uL)	Final Concentration (ug/mL)
8270 Mega Mix	Restek	31850	500/1000	500	166.67
Benzoic acid	STLVT	NA	20.000	24.9	166.67
Methylene Chloride	J.T. Baker	9266-95	NA	2475.1	NA
					Final Volume = 3000 uL

SOM01.1 Added Compound Mix

Component	Vendor	Catalog #	Concentration (ug/mL)	Volume Added (uL)	Final Concentration (ug/mL)
CLP SV SOW DMC	Cambridge Isotope	ES-5037	2000	600	1000
1,2,4,5-Tetrachlorobenzene	Lab Prepared	NA	20000	60	1000
Methylene Chloride	J.T. Baker	9266-95	NA	540	NA
					Final Volume = 1200 uL

SOM01.1/OLM04 Added Compound Mix

Component	Vendor	Catalog #	Concentration (ug/mL)	Volume Added (uL)	Final Concentration (ug/mL)
OLM04 SV Mix	SUPELCO	47514-U	2000	1300	1000
Methylene Chloride	J.T. Baker	9266-95	NA	1300	NA
					Final Volume = 2600 uL

SOM01.1 Internal Standard Solution

Component	Vendor	Catalog #	Concentration (ug/mL)	Volume Added (uL)	Final Concentration (ug/mL)
SV Internal Standard Mix	Restek	31006	4000	1300	500
Methylene Chloride	J.T. Baker	9266-95	NA	9100	NA
					Final Volume = 10400 uL

SOM01.1 Benzidines Mix

Component	Vendor	Catalog #	Concentration (ug/mL)	Volume Added (uL)	Final Concentration (ug/mL)
Benzidines Mix	Ultra Scientific	US-105N	2000	500	1000
Methylene Chloride	J.T. Baker	9266-95	NA	500	NA
					Final Volume = 1000 uL

SOM01.1 DFTPP Solution

Component	Vendor	Catalog #	Concentration (ug/mL)	Volume Added (uL)	Final Concentration (ug/mL)
GCMS Tune Standard	Ultra Scientific	GCM-160A	1000	400	25
Methylene Chloride	J.T. Baker	9266-95	NA	15600	NA
					Final Volume = 16000 uL

DMC Spike Solution

Component	Vendor	Catalog #	Concentration (ug/mL)	Volume Added (uL)	Final Concentration (ug/mL)
CLP SVOA DMC's	Cambridge isotope Labs	ES-5037	2000	5000	80ppm
Methanol	J.T. Baker	9263-03	NA	120000	NA
					Final Volume =125000 uL

Benzoic Acid

Component	Vendor	Catalog #	Concentration (ug/mL)	Volume Added (ug)	Final Concentration (ug/mL)
Benzoic acid	Aldrich	242381	neat	68.5	20000
Methylene Chloride	J.T. Baker	9266-95	NA	3425 uL	NA
					Final Volume = 3425 uL

1,2,4,5-Tetrachlorobenzene

Component	Vendor	Catalog #	Concentration (ug/mL)	Volume Added (ug)	Final Concentration (ug/mL)
1,2,4,5-Tetrachlorobenzene	Aldrich	131857	neat	60.8	20000
Methylene Chloride	J.T. Baker	9266-95	NA	3040ul	NA
					Final Volume =3040 uL

Routine SOM01.1 Working Calibration Standard Preparation

Sample Type	Final Concentration[1] (ng/uL)	CAL MIX (uL)	CAL MIX ADDED (uL)	BENZIDINES (uL)	ISTD (uL)	MeCl2 (uL)	Final Volume (uL)
CAL Level 1	10	3	0.5	0.5	4	96	104
CAL Level 2	20	6	1	1	4	92	104
CAL Level 3	40	12	2	2	4	84	104
CAL Level 4	80	24	4	4	4	68	104
CAL Level 5	160	48	8	8	4	36	104

[1] based on 2 uL injection volume

SIM SOM01.1 Working Calibration Standard Preparation

Sample Type	Final Concentration[1] (ng/uL)	CAL MIX (uL)	ISTD (uL)	MeCl2 (uL)	Final Volume (uL)
CAL Level 1	0.20	5	4	95	104
CAL Level 2	0.40	10	4	90	104
CAL Level 3	0.80	20	4	80	104
CAL Level 4	1.60	40	4	60	104
CAL Level 5	2.0	50	4	50	104

[1] based on 2 uL injection volume

Appendix B: Equations

Equation 1: Relative Response Factor Calculation

$$RRF = \frac{A_x}{A_{is}} X \frac{C_{is}}{C_X}$$

Where:
A_X = Area of the characteristic ion for the compound to be measured.
A_{is} = Area of the characteristic ion for specified internal standard.
C_{is} = Amount of the internal standard injected (ng).
C_x = Amount of the target compound or DMC injected (ng).

Equation 2: Percent Relative Standard Deviation

$$\% \text{ RSD} = \frac{Standard\ Deviation}{Mean} X100$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (X_i - \overline{X})^2}{(n-1)}}$$

Where:

X_i = Each individual value used to calculate the mean.
$\overline{X}$ = The mean of n values.
n= The total number of values.

Equation 3: Relative Response Factor Percent Difference

$$\%Difference_{RRF} = \frac{RRF_C - \overline{RRF_i}}{\overline{RRFi}} X100$$

Where:
$\overline{RRF}$ = Mean Relative Response Factor from the most recent initial calibration meeting technical acceptance criteria

RRF_c = Relative Response Factor from CCV standard.

Equation 4: Concentration of Water Samples

$$\text{Concentration } \mu\text{g/L} = \frac{(A_x)(I_s)(V_t)(Df)(GPC)}{(A_{is})(\overline{RRF})(V_o)(V_i)}$$

Where:
A_x = Area of the characteristic ion for the compound to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
I_s = Amount of internal standard injected in nanograms (ng).
V_o = Volume of water extracted in milliliters (mL).
V_i = Volume of extract injected in microliters (uL).
V_t = Volume of the concentrated extract in microliters (uL)
$\overline{RRF}$ = Mean Relative response factor determined from the initial calibration standard.

$GPC = \frac{Vin}{Vout}$ =GPC factor. (If no GPC is performed, GPC = 1).

Vin = Volume of extract loaded onto GPC column.
Vout = Volume of extract collected after GPC cleanup.
If no dilution is performed, Df = 1.0

Equation 5: Concentration of Soil and Sediment Samples

$$\text{Concentration } \mu\text{g/Kg (Dry weight basis)} = \frac{(A_x)(I_s)(V_t)(Df)(GPC)}{(A_{is})(RRF)(V_i)(W_s)(D)}$$

Where:
A_x, I_s, $A_{is,}$ Vin, and Vout are as given for water, above.
V_t = Volume of the concentrated extract in microliters (uL)
V_i = Volume of the extract injected in microliters (uL)

$$D = \frac{100 - \%\,\text{Moisture}}{100}$$

W_s = Weight of sample extracted in grams (g)
GPC = $\frac{Vin}{Vout}$ = GPC factor
RRF =Mean Relative Response Factor determined from the initial calibration standard.
If no dilution is performed, Df=0

Appendix C: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Concentration Level (low or medium): characterization of soil samples or sample fractions as low concentration or medium concentration is made on the basis of the laboratory's preliminary screen, not on the basis of information entered on the Traffic Report (C-O-C) by the sampler.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Decafluorotriphenylphosphine (DFTPP): compound chosen to establish mass spectral instrument performance for semivolatile analysis.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Extractable: a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (BNA) and pesticide/Aroclor compounds.

GEL Permeation Chromatography (GPC): a size-exclusion chromatographic technique that is used as a cleanup procedure for removing large organic molecules, particularly naturally occurring macro-molecules such as lipids, polymers, viruses, etc.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): solution prepared from a separate source from that which is used to prepare the calibration curve.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Internal Standards: compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample at a know concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

Isotopic Dilution: a process whereby the determination of the concentration of target analytes is based upon the recovery of spiked analogues.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix: the predominant material of which the sample to be analyzed is composed. For the purpose of this SOW, a sample matrix is either water, soil/sediment, or tissue. Matrix is not synonymous with phase (liquid or solid).

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Effect: in general, the effect of a particular matrix (water or soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles, which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike (MS): a field sample to which a known amount of specific target analytes are added.

Matrix Spike Duplicate (MSD): a field sample to which a known amount of specific target analytes are added as a duplicate of Matrix Spike.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Relative Response Factor (RRF): a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by

analysis of standards and are used in the calculation of concentrations of analytes in samples. See Section 12.0 Calculations.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Selective Ion Monitoring (SIM): a mass spectrometer acquisition mode in which the instrumentation is configured to selectively scan and acquire only those ions necessary for identification and quantitation of the target compounds. The limited mass range enables greater dwell times per mass than can be accomplished with full scan mode thereby enabling greater instrument sensitivity.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Deuterated Monitoring Compound(DMC): for semivolatiles, added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. DMCs are isotopically labeled compounds not expected to be detected in environmental media.

STANDARD OPERATING PROCEDURE
STL BURLINGTON

DETERMINATION OF PESTICIDES BY GC/ECD
CLP SOW SOM01.1

Applicable Matrix: Non-Potable Water, Soil & Sediment

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the analytical procedure used to determine the presence and concentration of the chlorinated pesticides found in water, soil and sediment samples collected from hazardous waste sites. This procedure is based on CLP SOW SOM01.1, Exhibit D Pesticides, USEPA Contract Laboratory Program Statement of Work for Organic Analyses, Multi-Media, Multi-Concentration, October 2004. This SOP can be used to determine analyte concentrations that range from the CLP Contract Required Quantitation Limits (CRQL) to one million times the CRQL in these matrices when appropriate dilutions are made.

1.2 The target compound list is provided in Table 1, Section 18.0, along with the Contract Required Quantitation Limit (CRQL). Recovery and precision limits for the laboratory control sample (LCS) and matrix spikes (MS/MSD) are provided in Table 3A and Table 3B, Section 18.0.

There are two isomers of heptachlor epoxide, the endo isomer (Isomer A) and the exo isomer (Isomer B). The two isomers are separable using current GC capillary columns, but only the exo isomer (Isomer B) is of environmental significance. Isomer B is the isomer used as an analytical standard, and it is the isomer identified, quantitated in sample analysis and reported on the appropriate forms as heptachlor epoxide.

2.0 SUMMARY OF METHOD

2.1 Water and soil/sediment samples are extracted and cleaned up following the procedures given in laboratory SOP LM-OP-SOM01.1-Pest. After cleanup, extracts are analyzed using a dual column Gas Chromatograph / Electron Capture Detector (GC/ECD).

3.0 DEFINITIONS

3.1 A list of terms and definitions is given in Appendix C.

4.0 INTERFERENCES

4.1 Method Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment. These contaminants can lead to elevated baselines and discrete artifacts that appear in gas chromatograms. These materials are demonstrated to be free of interferences by the analysis of method blanks. Interferences from phthalate esters can also pose a problem for the chlorinated pesticides analysis. To avoid this type of interference, the use of plastics in the laboratory must be minimized.

4.2 Matrix Interferences

Matrix interferences may be caused by contaminants that are co-extracted with the

samples. The extent of the interference will vary by matrix and are minimized using the various cleanup procedures described in this SOP.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 Specific Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

Table 2, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

Brand names and/or catalog numbers provided in this section are subject to change.

6.1 Gas Chromatograph / Electron Capture Detector (GC/ECD System)

- Hewlett Packard 6890.
- Hewlett Packard 7673 Autosampler, capable of a 2 uL injection.

6.2 GC Columns

- RTX - CLPesticides (30m x 0.32 mm ID x 0.25um), Restek # 11139.
- RTX - CLPesticides II, (30m x 0.32 mm ID x 0.25um), Restek # 11324.

6.3 Data System

- Data Acquisition: Multichrom Version 2.0.
- Data Processing and Report Generation: Target Version 3.5.

6.4 Miscellaneous

- Autosampler Vials, National Scientific.
- Volumetric Syringes, Class “A” (10uL, 25uL, 50uL, 100uL, 250uL and 500uL), Hamilton.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Hexane - pesticide quality -JT Baker.

Acetone - pesticide quality -JT Baker.

7.2 Standards

Stock standard solutions are purchased pre-made from commercial vendors. The laboratory prepares intermediate and working standards from stock standard solutions by diluting a known volume of stock standard in an appropriate solvent to a specified volume. The instructions for the preparation of intermediate and working standard solutions used in this procedure are provided in Appendix A.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 Unless otherwise specified by client or regulatory program, sample extracts are stored at a temperature of 4ºC (±2 ºC) for 30 days after delivery of the complete reconciled data package and then disposed of in accordance with applicable regulations. Samples, sample extracts, and standards are stored separately.

8.2 The analytical holding time is 40 days from the start of extraction.

9.0 QUALITY CONTROL

9.1 A method blank (MB) and laboratory control sample (LCS) are prepared with each extraction batch. Matrix spikes (MS/MSD) are prepared with each extraction batch of every 20 or fewer field samples when sufficient sample volume is available. Sample duplicates (SD) and/or client-specific MS/MSD are performed per client request. The method blank is analyzed on each GC/ECD system used to analyze associated samples.

9.2 Surrogate compounds tetrachloro-meta-xylene (TCX) and decachlorobiphenyl (DCB) are added to all field and QC samples prior to extraction, and are added to the instrument blanks.

9.3 All extracts undergo sulfur cleanup in the extraction procedure. At times it is necessary to do an additional sulfur cleanup on one or more of the extracts. When part of a set of samples is subjected to additional sulfur cleanup, a sulfur cleanup blank (PSBLK##) is prepared and analyzed in order to determine the level of contamination associated with the sulfur cleanup procedure.

9.4 Instrument blanks (PIBLK##) are analyzed during the analytical sequence to determine the level of contamination associated with the instrumental analysis.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Set-Up and Operating Conditions

Install a five meter deactivated guard column to the injection port, and using a glass "Y" connect the guard column to the separate analytical columns. Attach the analytical columns to each Electron Capture Detector (ECD).

The following conditions are recommended unless otherwise noted:

Carrier Gas:	Hydrogen (Hydrogen Generator)
Column Flow:	2.0 mL/min.
Make-up Gas:	Argon/Methane
Make-up Gas Flow:	120 mL/min.
Injector Temperature:	225°C
Detector Temperature	300°C
Injection Technique:	on-column
Injection volume:	2 µL
Injector:	CTC autosampler
Initial Temperature:	120°C
Initial Hold Time:	1 minute
Temperature Program:	16°C /min. to 210°C 6°C /min. to 245°C 12.5°C/min to 300°C. Hold for 4 minutes.

10.2 Initial Calibration

Calibrate the GC using five different concentrations of the single component pesticides, Toxaphene and the surrogates in order to determine the instrument's sensitivity and linearity. The recommended concentration for each calibration level is provided in Appendix A. Perform initial calibration upon initial award of the contract, whenever major instrument maintenance or modification is performed (for example, column replacement, baking or replacement of ECD), or if the calibration verification technical acceptance criteria are not met.

10.2.1 Calibration Procedure.

Set up the GC/ECD system as described in Section 10.1. Prepare the calibration standards using the instructions provided in Appendix A. Allow the standards to warm to ambient temperature prior to analysis. Two calibration sequence options are provided in CLP SOM01.1. The laboratory has chosen to calibrate pesticides using the single mixture (INDC). Analyze the standards in the initial calibration sequence below.

Calibration Sequence (CLP SOM01.1 Sequence 1)

Calibration Standard	EPA Sample Number
Instrument Blank	PIBLK##
Resolution Check	RESC##
Performance Evaluation Mixture (PEM)	PEM##
Toxaphene CS1	TOXAPH1##
Toxaphene CS2	TOXAPH2##
Toxaphene CS3	TOXAPH3##
Toxaphene CS4	TOXAPH4##
Toxaphene CS5	TOXAPH5##
CS1 Individual Standard Mix C	INDC1##
CS2 Individual Standard Mix C	INDC2##
CS3 Individual Standard Mix C	INDC3##
CS4 Individual Standard Mix C	INDC4##
CS5 Individual Standard Mix C	INDC5##
Instrument Blank	PIBLK##
PEM	PEM##

During the initial calibration the data system measures the RT of each single component pesticide for each of the five calibration standards (Individual Standard Mix C). For Toxaphene, it measures the RTs for the 5 quantitation peaks chosen. Calculate the mean absolute RT ($\overline{RT}$) for each single component pesticide, surrogate and Toxaphene quantitation peaks as the average of the five values. The RT window is calculated for each single component pesticide, surrogate and Toxaphene quantitation peaks using the RT windows given in Table 1, Section 18.0.

The data system determines the linearity of the instrument by calculating the Percent Relative Standard Deviation (%RSD) of the Calibration Factors (CFs) for all single component pesticides, surrogates and the five quantitation peaks chosen for Toxaphene. The laboratory's preferred method to calculate the CF is peak height but the SOW allows the CF to be calculated from peak height or peak area. For instance, the SOW specifies that the linearity of one individual component may be calculated in peak area and another individual component may be calculated in peak height. For example, it is acceptable to calculate the linearity of DDT using peak height and to calculate the linearity of DDD using peak area. It is also permitted to choose peak area for all quantitation peaks chosen for Toxaphene. However, the SOW does not allow the

calculation of the CF for one individual component in peak area and height. For example, you cannot calculate the CF for the CS1 standard of Aldrin in peak height and calculate the CF for CS3 of Aldrin in peak area. For Toxaphene, it is not permitted to calculate the CF of peak 1 in peak height and peak 4 in peak area. Whether peak area or height is used, the option chosen must remain consistent for all calculations for that analyte.

The data system calculates the breakdown of DDT, the breakdown of endrin and the combined breakdown in the PEM. The data system calculates the percent difference for each pesticide and surrogate in the PEM. The resolution between the analytes in the Resolution Check, PEM, and CS3 Individual Standard Mixtures are calculated and entered into the data processing system. Equations for the calculation of the calibration factor (CF), mean calibration factor, Percent Relative Standard Deviation, percent breakdown, percent difference and resolution are provided for reference in Appendix B.

10.2.2 Acceptance Criteria for Initial Calibration

The acceptance criteria described in this section apply independently to each GC column. The acceptance criteria must be met prior to sample analysis. If criteria are not met, identify and correct the problem prior to further analysis.

- The initial calibration sequence must be analyzed in the order specified in Section 10.2.1.
- The resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 80.0% for all analytes on both columns.
- All single component pesticides and surrogates in the PEMs must be greater than or equal to 90.0% resolved on each column.
- The absolute RTs of the single component pesticides and surrogates in the PEMs must be within the established RT window.
- The Percent Difference between the calculated amount and nominal amount for each of the single component pesticides and surrogates in the PEMs must be greater than or equal to –25.0% and less than or equal to 25.0%.
- The percent breakdown of DDT and Endrin in PEMs must be less than or equal to 20.0%. The combined breakdown of DDT and Endrin must be less than or equal to 30.0%.
- The %RSD of the CFs for each single component pesticide must be less than or equal to 20.0%, except for alpha-BHC and delta-BHC. The %RSD for alpha-BHC and delta-BHC must be less than 25.0%. The %RSD of the CFs for TCX and DCB (surrogates) must be less than 30.0%. Up to two single component pesticides (not surrogates) per column may exceed 20.0% RSD (25.0% for alpha-BHC and delta-

BHC) but they must be less than 30.0% RSD. The %RSD of the CFs for Toxaphene must be less than or equal to 30.0%.

- The resolution between any two adjacent peaks in the CS3 Individual Standard Mixture C must be at least 80.0% on both columns.

- All instrument blanks must meet the following criteria:
  - The instrument blank must be analyzed at the original concentration.
  - The instrument blank must meet the acceptance criteria for sample analysis.
  - The concentration of each target analyte in the instrument blanks must be less than the CRQL on each chromatographic column.
  - The RT for each surrogate must be within the established RT window.

10.3 Calibration Verification

Three types of analyses are used to verify the calibration and evaluate instrument performance. They are instrument blanks (PIBLKs), PEMs and the CS3 Individual Standard Mixtures. Calibration Verification consists of a PIBLK and PEM or a PIBLK and the CS3 Individual Standard Mixture. Sample data must be bracketed by acceptable analyses of instrument blanks, PEMs and the Individual Standard Mixture.

10.3.1 Frequency of Calibration Verification

Bracket one end of a 12-hour period during which data for field and QC samples are collected with a PIBLK and PEM standard. Bracket the other end of the 12-hour period with a second PIBLK and Individual Standard Mix CS3.

For the 12-hour period immediately following the initial calibration sequence, the PIBLK and the PEM that are analyzed in the last two steps of the initial calibration sequence may bracket the front end of the 12-hour period. The injection of the PIBLK begins the 12-hour period. Inject a PIBLK and the Individual Standard Mix CS3 immediately after the 12-hour period. Continue to use this progression until any of the technical acceptance criteria for the PIBLKs, PEMs or Individual Standard Mix CS3s fail.

If the instrument sits idle for more than 12 hours, analyze a PIBLK and PEM in order to start a new sequence.

No more than 14 hours may elapse from the injection beginning the opening CCV and the injection of the closing CCV. If Toxaphene is detected in a sample, analyze a Toxaphene CCV standard within 72 hours of the analyte detection in the sample and in a valid 12-hour period.

10.3.2 Procedure for Calibration Verification

Allow the standards to warm to ambient temperature prior to analysis. Analyze the PIBLK, PEM and CS3 Individual Standard Mixture at the required frequencies.

10.3.3 Acceptance Criteria for Calibration Verification

The acceptance criteria listed below applies independently to each column. The acceptance criteria must be met prior to analysis of field and QC samples.

- The PEMs, Individual Standard Mixture and PIBLKs must be analyzed at the required frequency.

- All single component pesticides and surrogates in the PEMs used to demonstrate calibration verification must be greater than or equal to 90.0% resolved. The resolution between any two adjacent peaks in the Individual Standard Mix CS3 must be at least 80.0% on both columns.

- The absolute RT for the single component pesticides and surrogates in the PEMs and CS3 Individual Standard Mixture must be within the RT windows.

- The percent difference between the calculated amount and nominal amount for each single component pesticide and surrogates in the PEM used to demonstrate calibration verification must be greater than or equal to –25.0% and less than or equal to 25.0%.

- The percent breakdown of DDT and Endrin in the PEM must be less than or equal to 20.0% on each column. The combined breakdown must be less than or equal to 30.0% on each column.

- The percent difference between the CF of each single component pesticide and surrogates in the Individual Standard Mixture CS3, and the mean calibration factor from the initial calibration must be greater than or equal to –20.0% and less than or equal to 20.0%. The percent difference between the CF of each quantitation peak of Toxaphene and the mean calibration factor from the initial calibration must be greater than or equal to –20.0% and less than or equal to 20.0%.

- All instrument blanks must meet the following criteria:
  - The instrument blank must be analyzed at the original concentration.
  - The instrument blank must meet the acceptance criteria for sample analysis.
  - The concentration of each target analyte in the instrument blanks must be less than the CRQL on each chromatographic column.
  - The RT for each surrogate must be within the established RT window.

Equations for the calculation of the calibration factor (CF), mean calibration factor, relative standard deviation, percent breakdown, percent difference and resolution are provided for reference in Appendix B.

11.0 PROCEDURE

Before field and QC samples and required blanks can be analyzed, ensure that the instrument meets criteria for initial calibration and calibration verification. Analyze all

samples and standards under the same instrument conditions (See Section 10.1) using the same on-column injection volume of 2.0 uL.

11.1 Extract Screening and Guidance for Dilution

Prepare the extracts at a 1:10 dilution for screening. Screen all extracts on a designated screen instrument prior to instrument analysis to determine the presence of sulfur and to assess target analyte concentrations. Use the results of the screen to determine the approximate dilution factor (DF) required to get the largest peak within the calibration range. If sulfur is found in the extract, repeat sulfur cleanup following the procedure given in laboratory SOP LM-OP-SOM1.1-PEST.

- If the response of any single target analyte is greater than the response of the high standard (CS5) of the initial calibration range on **both** columns, dilute the extract such that the response of the pesticide compounds in the diluted extract is between the low standard (CS1) and the high standard (CS5) for the lower column response of the two analyses.

- If the response of any Toxaphene peak used for quantitation is greater than the response of the corresponding Toxaphene peak in the high standard (CS5), dilute the extract so that the response of the same peak is between the mid standard (CS3) and the high standard (CS5).

- If a dilution is performed solely to bring a peak within calibration range or to get the Toxaphene pattern on scale, report the results for both the greater and the less concentrated extracts.

- If the dilution factor is greater than 10, analyze the extract 10 times more concentrated than the diluted sample extract and report both sets of data. If the dilution factor is less than 10, analyze the extract undiluted and report both sets of data.

- If the response of any target analyte is above the high calibration standard (CS5) after a dilution of 1:100,000 contact the project manager for further instruction.

11.2 Analysis

Analyze the samples using the following analytical sequence:

Injection #	Material Injected
1-12	First 12 steps of initial calibration sequence 1 (See Section 10.2.1)
13	PIBLK at end of initial calibration sequence 1
14	PEM at end of initial calibration sequence 1
15	First sample if using initial calibration sequence 1
1st injection past 12 hours	PIBLK

2nd injection past 12 hours	Individual Standard Mixture C

The above analysis sequence shows only the minimum required standards. More standards may be run at the discretion of the laboratory. However, the standards must satisfy all criteria in order to continue the sequence.

Load the extracts and standards into the autosampler and enter the sample ID's into the data acquisition program in the same order that the extracts and standards were placed in the autosampler. Start the analytical sequence and acquire the data.

The data system tentatively identifies target analytes by comparing the retention time of the peaks to the retention time windows established in the intial calibration. Single component analytes are identified when peaks are observed in the RT window for the analyte on both GC columns. The data system uses a set of 3-5 major peaks for Toxaphene. RT windows for each peak are determined in the initial calibration. Identification of Toxaphene in the sample is based on pattern recognition and if the 3-5 major peaks chosen are within the established RT windows on both GC columns. If Toxaphene is detected in a sample, analyze a Toxaphene CS3 standard in a valid analytical sequence within 72 hours of detection in the sample. If the data system does not properly integrate a peak, perform and document manual integration in accordance with the laboratory SOP for *Manual Integration*.

The data system performs quantitation for all analytes (including Toxaphene) and surrogates for each GC column based on the mean calibration factor from the most recent initial calibration. Additionally, the quantitation of Toxaphene is accomplished by comparing the height or area (See section 10.2.1) of each of the 3-5 major peaks in the sample with the same peaks from initial calibration. The concentration of each peak is determined then a mean concentration is determined for each column. The lower of the two concentrations (between columns) is reported on the Form I.

The data system adjusts calculated sample results to account dilution and percent moisture. The data system also calculates the percent recovery for the surrogates for each GC column as well as the relative percent difference between results on each GC column. Although the data system performs the calculations, equations for the calculation of sample concentration is provided for reference in Appendix B.

Assess the results to ensure acceptance criteria are met. The acceptance criteria for QC samples are summarized in Section 18.0, Table 4. The acceptance criteria for field samples are listed below. If criteria are not met, perform corrective action. Corrective action may include qualification of data, instrument maintenance, or re-extraction and reanalysis.

- Samples (field and QC) and standards were analyzed under the instrument operating conditions provided in Section 10.1

- If the samples required GPC cleanup, the GPC system used must have met the acceptance criteria for calibration and verification as specified in laboratory SOP LM-OP-SOM01.1-PEST.

- Samples were cleaned-up using Florisil that met the technical acceptance criteria for Florisil cartridge lot verification as specified in laboratory SOP LM-OP-SOM01.1-PEST.

- The samples were extracted and analyzed within holding time.

- Samples were associated with a method blank and an LCS that met acceptance criteria.

- The method blank was analyzed on each instrument on which associated samples were analyzed. Additionally, the method blank was subject to sulfur cleanup along with the associated samples, or a sulfur cleanup blank was performed. The surrogate recovery for the method blank was between 30-150%.

- The RT for each surrogate is within the RT window.

- The percent recovery for each surrogate in samples is within 30-150%*. **Up to one surrogate may fail criteria per column and the surrogate recovery limits are not applicable to samples that were diluted.*

- No target responses exceed the upper limit of the calibration curve unless a dilution for the exceeding response was also analyzed.

- A Toxaphene standard CS3 was analyzed within 72 hours of detection of Toxaphene in any field sample.

If a target analyte concentration (on column) is greater than or equal to 5.0 ng/uL for both columns, contact the project manager to determine whether or not GC/MS confirmation is required.

If the percent difference of the concentrations between the 2 columns is greater than 25%, flag the results with a “P” data qualifier.

11.3 Data Reporting & Order of Deliverables

Evaluate each chromatogram to verify qualitative and quantitative identification of target compounds. Exhibit B of the CLP SOW specifies that the laboratory report estimated values qualified with a “J” data qualifier when the retention time (RT) data indicate the presence of a compound that meets the identification criteria and the result is less than the CRQL but greater than zero. For instance, is the sample quantiation limit is 5.0 ug/L but a concentration of 3.0 ug/L is calculated, the laboratory should report the results as 3.0J. However, the laboratory is further instructed to not report the estimated value if the presence of the peak is considered to be from instrument noise or other interference.

Generate a report that is legible, labeled and complete in CLP SOW Format. Forms and data should be arranged in chronological order by column. Sample data should be arranged in increasing order by Client ID.

12.0 CALCULATIONS

12.1 See Appendix B.

13.0 DATA ASSESSMENT, CORRECTIVE ACTION & REPORTING

13.1 Data Review and Corrective Action

Review all samples, standards, and QC samples against the acceptance criteria provided in this SOP. If the results do not fall within the criteria, perform corrective action. If corrective action is not taken, or is unsuccessful, document the situation with a nonconformance report and qualify the data using an appropriate data qualifier (See Appendix C for data qualifier definitions). For additional guidance regarding the laboratory's protocol and required elements for each level of data review (primary, secondary, and tertiary) refer to laboratory SOP LP-QA-003 *Data Review*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) must be determined on each instrument used for analysis for each matrix prior to initial use of the method. The MDLs are then verified annually with an MDL Verification Check sample that is subject to the same extraction and cleanup procedures as used for field samples. Some regulatory programs may require that the MDL study be repeated annually, but this is not a requirement of the CLP SOW. The procedure and acceptance criteria for MDL studies and MDL verficiation are given in laboratory SOP LP-QA-005 *Method Detection Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 Waste Streams Produced by the Method

The following waste streams are produced when this procedure is performed:

- Vials containing sample extracts

Transfer the waste stream(s) to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the

satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in laboratory SOP LP-LB-001 *Hazardous Waste*.

16.0 REFERENCES

16.1 CLP SOW SOM01.0 Multi-Media, Multi-Concentration Statement of Work for Organics Analysis, October 2004.

16.2 CLP SOW Summary of Changes SOM01.0 (10/08/04) to SOM01.1 (05/26/05).

17.0 SOP REVISION HISTORY

17.1 Added Revision History section.
17.2 Changed Title page to reflect current management structure.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Target Compound List, CRQL, RT Windows
18.2 Table 2: Primary Materials Used
18.3 Table 3A: Control Limits for LCS
18.4 Table 3B: Control Limits for MS/MSD
18.5 Table 4: QC Summary and Recommended Corrective Action
18.6 Appendix A: Standard Preparation Tables
18.7 Appendix B: Equations
18.8 Appendix C: Terms and Definitions

Table 1: Target Compound List, CRQL, and RT Windows

Analyte	Contract Required Quantitation Limit		Retention Time Windows
	Water ug/L	Solid ug/Kg	
alpha-BHC	0.050	1.7	±0.05
beta-BHC	0.050	1.7	±0.05
delta-BHC	0.050	1.7	±0.05
gamma-BHC (Lindane)	0.050	1.7	±0.05
Heptachlor	0.050	1.7	±0.05
Aldrin	0.050	1.7	±0.05
Heptachlor epoxide	0.050	1.7	±0.07
Endosulfan I	0.050	1.7	±0.07
Dieldrin	0.10	3.3	±0.07
4,4-DDE	0.10	3.3	±0.07
Endrin	0.10	3.3	±0.07
4,4-DDD	0.10	3.3	±0.07
Endosulfan II	0.10	3.3	±0.07
Endosulfan sulfate	0.10	3.3	±0.07
4,4-DDT	0.10	3.3	±0.07
Methoxychlor	0.50	17	±0.07
Endrin ketone	0.10	3.3	±0.07
Endrin aldehyde	0.10	3.3	±0.07
alpha-Chlordane	0.050	1.7	±0.07
gamma-Chlordane	0.050	1.7	±0.07
Toxaphene	5.0	170	±0.07
TCX (Surrogate)	NA	NA	±0.05
DCB (Surrogate)	NA	NA	±0.10

Table 2: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 3A: Laboratory Control Sample Recovery Limits

Analyte	Recovery Limits (%R)
	Water/Soil
gamma-BHC	50-120
Heptachlor epoxide	50-150
Dieldrin	30-130
4,4'-DDE	50-150
Endrin	50-120
Endosulfan sulfate	50-120
gamma-Chlordane	30-130

Table 3B: MS/MSD Recovery and Precision Limits

Analyte	Recovery Limits (%R)	RPD	Recovery Limits (%R)	RPD
	Water	Water	Solid	Solid
gamma-BHC	56-123	0-15	46-127	0-50
Heptachlor	40-131	0-20	35-130	0-31
Aldrin	40-120	0-22	34-132	0-43
Dieldrin	52-126	0-18	31-134	0-38
Endrin	56-121	0-21	42-139	0-45
4,4'-DDT	38-127	0-27	23-134	0-50

Table 4: QC Summary and Recommended Corrective Action

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[1]
Initial Calibration	Initially, prior to sample analysis; when calibration verification standards indicate calibration relationship is no longer valid; after major instrument maintenance	See Section 10.2	Correct problem and repeat initial calibration.
Calibration Verification	To open and close 12 hour sample windows.	See Section 10.3	Correct problem, reanalyze CCV and all associated samples since last successful CCV
Method Blank	One per extraction batch (Not to exceed 20 field samples) Bracketed by 12 hour windows	Target analytes must be < CRQL Surrogate recovery: 30-150% [Not advisory] Analyzed on each GC/ECD system used to analyze field samples. RT of surrogates must be within RT windows	Correct problem, re-extract and reanalyze with all associated samples
Sulfur Blank	Whenever a subset of samples within an extraction batch receives sulfur clean up.	Target analytes must be < CRQL Surrogate recovery: 30-150% [Not advisory] Analyzed on each GC/ECD system used to analyze field samples. RT of surrogates must be within RT windows	Correct problem, re-extract and reanalyze with all associated samples.
Instrument Blank	First analysis in 12-hour analytical sequence; preceding CCVs Preceding and following acceptable sample analyses.	Target analytes must be < CRQL Analyzed on each GC/ECD system used to analyze field samples. RT of surrogates must be within RT windows Requirements apply independently to each column	Correct problem and reanalyze with all associated samples.
LCS	One per extraction batch	%R within control limits given in Table 3A Surrogate recovery: 30-150% [Not advisory] RT of surrogates must be within RT windows Requirements apply independently to each column	Correct problem, re-extract and reanalyze with all associated samples.
MS/MSD	One per sample set of 20 field samples analyzed at the most concentrated analysis of parent. Note: Results can be reported from over-range response.	%R & %RPD within advisory limits given in Table 3B RT of surrogates must be within RT windows	Evaluate circumstances. Assay spike formulations.
Surrogate Spike	All field and QC samples	%R 30-150 for all field and QC samples *Up to one surrogate may fail criteria per column in samples.*	Correct problem, re-extract and reanalyze if sufficient sample is available

[1]*The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

Appendix A: Standard Preparation Tables

The formulations provided in this section may be subject to change. If the concentration or volume of any of the components change the formulation must be adjusted accordingly. Unless otherwise noted, an expiration date of 6 months from date of preparation is assigned to all intermediate and working standards. See laboratory SOP LP-LB-002 *Standard Preparation* for further guidance on the preparation of standard solutions.

Table Legend:
Cstock = Concentration of Parent Standard
Vstock = Volume of Parent Standard
Vspike = Volume of Prepared Standard
Cspike = Theoretical Concentration of Prepared Standard

INTERMEDIATE STANDARDS

Toxaphene Intermediate Standard solution (50 mg/L)

Stock Standard	*Cstock mg/L*	*Vstock mL*	*Vspike mL*	*Cspike mg/L*	*Solvent*
Toxaphene*	1000	1.0	20	50	Hexane

**Restek, Catalog #32005*

TCMX Intermediate Standard Solution (4 mg/L)

Stock Standard	*Cstock mg/L*	*Vstock uL*	*Vspike mL*	*Cspike mg/L*	*Solvent*
TCX*	2000	100	50	4.0	Hexane

**Ultra Catalog # IST-440*

DCB Intermediated Standard Solution(8 mg/L)

Stock Standard	*Cstock mg/L*	*Vstock mL*	*Vspike mL*	*Cspike mg/L*	*Solvent*
DCB*	200	1.0	25	8.0	Hexane

**Restek Catalog #32029*

CALIBRATION STANDARDS

Instrument Blank Surrogate Spike Solution

Parent Standard	*Cstock mg/L*	*Vstock uL*	*Vspike mL*	*Cspike ug/L*	*Solvent*
TCX Intermediate	4.0	500	100	20	Hexane
DCB Intermediate	8.0	1000		40	

Breakdown Check Standard

Parent Standard	*Cstock mg/L*	*Vstock uL*	*Vspike mL*	*Cspike ug/L*	*Solvent*
Pesticide Performance Evaluation Mix*	10-250	30	30	10-250	Hexane

**Ultra Catalog #CLP-250*

Individual Standard Mix CS5 (Single Component Pesticides)

Parent Standard	*Cstock mg/L*	*Vstock uL*	*Vspike mL*	*Cspike ug/L*	*Solvent*
Organochlorine Pest Mix AB#2*	8-80	1.0	100	80-800	Hexane
TCX Intermediate	4	2.0		80	
DCB Intermediate	8	2.0		160	

**Restek Catalog #32292*

Calibration Standards CS4-CS1 (Single Component Pesticides)

Parent Standard	Volume	CS4	CS3	CS2	CS1
CS5	V*stock* (mL)	20	10	5	2.5
	V*spike* (mL)	40	40	40	40

Dilute the volume of CS5 specified (v*stock*) in hexane to the volume specified (v*spike*).

Toxaphene Standard Mix CS5

Parent Standard	*Cstock mg/L*	*Vstock uL*	*Vspike mL*	*Cspike ug/L*	*Solvent*
Toxaphene Intermediate	50	16	100	8000	Hexane
TCX Intermediate	4	2		80	
DCB Intermediate	8	2		160	

Toxaphene Calibration Standards CS4-CS1

Parent Standard	Volume	CS4	CS3	CS2	CS1
CS5	V*stock* (mL)	20	10	5	2.5
	V*spike* (mL)	40	40	40	40

Dilute the volume of CS5 specified (v*stock*) in hexane to the volume specified (v*spike*).

Final Concentration of Prepared Calibration Standards in Extract

Analyte	CS1 (ng/mL)	CS2 (ng/mL)	CS3 (ng/mL)	CS4 (ng/mL)	CS5 (ng/mL)
TCX (surrogate)	5	10	20	40	80
DCB (surrogate) Pesticides	10	20	40	80	160
alpha-BHC	5	10	20	40	80
beta-BHC	5	10	20	40	80
delta-BHC	5	10	20	40	80
gamma-BHC	5	10	20	40	80
Heptachlor	5	10	20	40	80
Aldrin	5	10	20	40	80
Heptachlor epoxide	5	10	20	40	80
Endosulfan I	5	10	20	40	80
Dieldrin	10	20	40	80	160
4-4'-DDE	10	20	40	80	160
Endrin	10	20	40	80	160
Endosulfan II	10	20	40	80	160
4-4'-DDD	10	20	40	80	160
Endosulfan sulfate	10	20	40	80	160
4,4'-DDT	10	20	40	80	160
Methoxychlor	50	100	200	400	800
Endrin ketone	10	20	40	80	160
Endrin aldehyde	10	20	40	80	160
gamma-Chlordane	5	10	20	40	80
alpha-Chlordane	5	10	20	40	80
Toxaphene	500	1000	2000	4000	8000

APPENDIX B: EQUATIONS

These equations were taken from reference method SOM01.0 and from SW-846 8000B, Test Methods for Evaluation of Solid Waste Revision 2, December 1996. USEPA.

Resolution

$$\%\text{Resolution} = \frac{V}{H} \otimes 100$$

Where:

V Depth of valley between the two peaks. Measure the depth of the valley along vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks.

H Height of the shorter of the adjacent peaks.

Absolute Retention Times

$$\overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n}$$

Where:

$\overline{RT}$ Mean Absolute Retention Time of analyte
RT_i Absolute Retention Time of analyte
n Number of measurements

Percent Breakdown

$$\%\,\text{Breakdown of DDT} = \frac{\text{amount found (ng) (DDE} + \text{DDD)}}{\text{amount (ng) of DDT injected}} \otimes 100$$

$$\%\,\text{Breakdown of Endrin} = \frac{\text{amount found (ng) (endrin aldehyde} + \text{endrin ketone)}}{\text{amount (ng) of endrin injected}} \otimes 100$$

$$\text{Combined \% Breakdown} = \%\,\text{Breakdown DDT} + \%\,\text{Breakdown Endrin}$$

Calibration Factor (CF_i)

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass Injected (ng)}}$$

Mean Calibration Factor ($\overline{CF}$)

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where:

CF_i Calibration Factor
n Number of Calibration Standards

Standard Deviation of the Calibration Factor (SD)

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)_2}{n-1}}$$

Where:

CF_i Calibration Factor
$\overline{CFi}$ Mean Calibration Factor
n Number of calibration levels

Percent Relative Standard Deviation (RSD) of the Mean Calibration Factor

$$\%RSD = \frac{SD}{\overline{CF}} \otimes 100$$

Where:

SD Standard Deviation of Calibration Factors
$\overline{CF}$ Mean Calibration Factor

Percent Difference (%D)- Used for Calibration Verification Standards

$$\%D = \frac{CF_v - \overline{CF}}{\overline{CF}} \otimes 100$$

Where:

CF_v Calibration Factor from the Continuing Calibration Verification (CCV)

Percent Difference (%D)- Used for PEM standards

$$\%D = \frac{C_{calc} - C_{nom}}{C_{nom}} \otimes 100$$

Where:
C_{calc} Calculated concentration
C_{nom} Nominal concentration

Percent Difference (%D)- Used for Form X

$$\%D = \frac{Conc_H - Conc_L}{Conc_L} \otimes 100$$

Where:
$Conc_H$ Higher of the two concentrations
$Conc_L$ Lower of the two concentrations

Percent Recovery (%R)

$$\%R = \frac{C_s}{C_n} \otimes 100$$

Where:
C_s Concentration of the Spiked Field or QC Sample
C_n Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD

$$\%R = \frac{C_s - C_u}{C_n} \otimes 100$$

Where:
C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (RPD)

$$RPD = \frac{|MSR - MSDR|}{1/2(MSR + MSDR)} \otimes 100$$

Where:

MSR Matrix Spike Recovery
MSDR Matrix Spike Duplicate Recovery

Sample Concentration

Water

$$\text{Concentrat ion}_{\text{sample}}\,(\text{ug/L}) = \text{Amt}_{\text{extract}}\,(\text{ng/uL}) \otimes \frac{\text{extract volume (uL)}}{\text{sample volume (mL)}} \otimes \text{DF}$$

Solids

$$\text{Concentration}_{\text{sample}}\,(\text{ug/Kg}) = \text{Amt}_{\text{extract}}\,(\text{ng/uL}) \otimes \frac{\text{extract volume (uL)}}{\text{sample weight (g)}} \otimes \frac{100}{\%\,\text{solids}} \otimes \text{DF} \otimes \text{GPC}$$

Where:

$$\text{AMT}_{\text{extract}}\,(\text{ng/uL}) = \frac{\text{Peak Area (or Height)}}{\overline{\text{CF}}} \otimes \frac{1}{\text{Vi}}$$

$$\text{GPC} = \frac{\text{volume loaded on GPC}}{\text{volume extract collected after GPC}}$$

Vi Amount Extract
DF Dilution Factor, if no dilution performed DF=1.

Appendix C: Terms & Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Aliquot: A measured portion of a field sample, standard, or solution taken for sample preparation and/or analysis.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Breakdown: A measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Factor: A measure of the Gas Chromatographic response of a target analyte to the mass injected.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

The EPA-defined qualifiers that are routinely used for this test method are:

U: Compound analyzed for but not detected at a concentration at or above the reporting limit.

J: Estimated Value

- Result is less than the CRQL but above the adjusted MDL
- RT data indicates the presence of a pesticide that meets the identification criteria and the result is less than the CRQL but above zero.

P: There is greater than 25% difference for detected concentrations between two GC columns

C: Positive result whose identification has been confirmed by GC/MS

B: Compound is found in the sample and the associated method blank.

E: Compound whose response exceeds the upper limit of the calibration range.

D: Concentration identified from a dilution analysis.

X,Y,Z: Laboratory defined flags that may be used alone or combined as needed. If used, provide a description of the flag in the project narrative.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Instrument Blank: a blank designed to determine the level of contamination associated with the analytical instruments.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

ARCADIS BBL

Attachment 1-3

STL ILM Inorganic SOPs

STANDARD OPERATING PROCEDURE
STL BURLINGTON

CLP SOW ILM05.3 / ICP- AES

Applicable Matrices: Waters, Soil, Sediment and Sludge

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for inductively coupled atomic plasma-atomic emission spectroscopy (ICP-AES) used to analyze water, sediment, sludge, and soil samples taken from hazardous waste sites.

1.2 This procedure is applicable to the elements listed in Table 1, Section 18.0.

2.0 SUMMARY OF METHOD

2.1 Water and soil samples are treated with acids and digested to solubilize the metals present. The digestates are analyzed for trace metals by atomic emission spectroscopy in which the samples are nebulized and the aerosol that is produced is transported to a plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed and the intensities of the lines are monitored by a photosensitive device that generates signal(s) processed by a computer. Background correction is performed to compensate for variable background contribution to the spectra. Each metal is quantified at specific wavelengths of light emitted as the electrons relax to lower energy states. The sample is analyzed by multiple integrations (2) and the average integration is converted to a concentration from a calibration curve.

2.2 This procedure is based on CLP SOW ILM05.3, Exhibit D for ICP-AES.

3.0 DEFINITIONS

3.1 A list of definitions is given in Appendix B.

4.0 INTERFERENCES

4.1 Spectral Inteferences

Overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena, and/or background contribution from stray light from the high concentration elements are examples of spectral interference that may cause target concentrations to be artificially high. These effects are compensated by using computer correction of the raw data by monitoring and measurement of the interfering element and/or background correction adjacent to the analyte line.

4.2 Physical Inteferences

Change in viscosity and surface tension can cause significant inaccuracies especially in samples that contain high dissolved solids and/or acid concentrations. The use of a peristalitic pump or sample dilution will minimize these interferences.

4.3 Chemical Interferences

Chemical interferences such as molecular compound formation, ionization effects and solute vaporization effects are highly dependent on matrix type and specific analyte elements. These interferences are not typical with ICP-AES analysis but if observed, can be minimized by matrix matching, buffering the sample and careful selection of instrument operating conditions.

5.0 SAFETY

5.1. Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added. Protective clothing such as a lab coat, safety glasses and latex gloves must be worn while performing this procedure.

5.3. Primary Materials Used

Table 2, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure**.** A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers and part numbers are subject to change at the discretion of the l laboratory.

6.1 Inductively Coupled Plasma – Atomic Emission Spectrometer (ICP-AES); Thermo Jarrell-Ash Trace ICP-AES 61E Trace or equivalent.

6.2 Block Digester: Capable of reaching 95°C.

6.3 Volumetric Pipettes: Calibrated daily.

6.4 Class A Volumetric Glassware.

6.5 Top-Loading Balance.

6.6 Filter apparatus.

6.7 Ribbed Watch glasses.

6.8 Whatman No. 42 Filter Paper or equivalent.

6.9 Polyethylene Digestion Vessels with Volumetric Indicators.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent Water - ASTM Type II Water (ASTM D1193-77) or equivalent.

Hydrochloric Acid, Concentrated (specific gravity 1.19); reagent grade; JT Baker or equivalent

Hydrochloric Acid (1:1): Fill a carboy half full with reagent water and slowly add an equal volume of concentrated HCl. Swirl to mix.

Nitric Acid, Concentrated (specific gravity 1.41) Hydrochloric Acid; reagent grade; JT Baker or equivalent

Nitric Acid, (1:1): Fill a carboy half full with reagent water and slowly add an equal volume of concentrated HNO_3. Swirl to mix.

Hydrogen Peroxide (30%); Hydrochloric Acid; reagent grade; JT Baker or equivalent

7.2 Standards

Stock standard solutions are purchased from commercial vendors. Working, secondary dilution and mixed calibration standards are prepared from stock standards by diluting volumes of stock standards with acidified reagent water in volumetric flasks to obtain the appropriate concentration. The components and recommended concentrations for calibration, working and secondary dilution standards are given in Appendix A.

Expiration dates of multi-component prepared solutions are assigned according to expiration date of the parent standard. Standard solutions are prepared as needed or on the expiration date, whichever occurs first.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 All samples should be collected in glass or polyethylene containers. Water samples must be preserved with nitric acid to a pH of less than 2 immediately following collection. All samples must be iced or refrigerated to 4°C (±2°C) and maintained at that temperature until digestion.

8.2 Dissolved Metals

For the determination of dissolved elements, the sample must be filtered through a 0.45 µm pore diameter membrane filter at the time of collection or as soon as possible. Preserve the filtrate with nitric acid to pH <2 immediately following filtration.

8.3 Samples are stored protected from light and refrigerated at 4°C (± 2°C) from the time of receipt until 60 days after delivery of a reconciled data package to the client. After 60 days the samples are disposed of in a manner that complies with all applicable regulations. Sample digestates are stored for 365 days after delivery of the complete, reconciled data package.

8.4 The maximum holding time for analysis is 180 days from Validated Time of Sample Receipt (VTSR).

9.0 QUALITY CONTROL

9.1 Preparation Blank (PB)

A PB is included with each digestion batch or every sample delivery group whichever is more frequent. The PB consists of reagent water and is prepared using the same reagents, in the same volumes and at the same acid concentration as the sample solution used for analysis. The PB is carried through all steps of the digestion procedure and analyzed in the same manner as samples.

The concentration of target analyte(s) in the PB must be less or equal to the CRQL. If it is not, the lowest concentration of that analyte in the associated samples must be greater than or equal to 10 times the blank concentration or the blank and associated samples must be re-digested and re-analyzed. If the concentration of the blank is below the negative CRQL, then all samples reported below 10 times the CRQL associated with the blank are re-digested and re-analyzed.

9.2 Interference Check Sample (ICS)

To verify interelement and background correction factors for all elements and for all interferents, analyze ICS solutions at the beginning and end of each analytical run, but not before the initial calibration verification (ICV) standard and at a minimum frequency of once per 20 samples per analytical run. The ICS analysis should be immediately followed by the continuing calibration blank/verification (CCV/CCB).

The ICS consists of two solutions (ICSA/ICSAB) that are analyzed consecutively starting with the ICSA. The ICSA solution includes the interferents, and the ICSAB includes the analytes mixed with the interferents.

The results of the ICSA should be within ±2 times the CRQL of the analyte's true value or ±20% of the analyte's true value in the ICSA, whichever is greater assuming the true value is zero unless otherwise stated. For analytes with a CRQL less than 5000ug/L, the ICSA results should be reported from the undiluted sample analysis. The results of the ICSAB

solution should be within ±2 times the CRQL of the true value or ±20% of the analyte's true value, whichever is greater. If the results are not within control limits, the analysis is stopped and the problem is investigated and corrected. The instrument is recalibrated and all samples analyzed since the last compliant ICSA/ICSAB are reanalyzed.

9.3 Laboratory Control Sample (LCS)

A laboratory control sample is prepared and analyzed in the same manner as samples with each digestion batch or every group of samples in a SDG, whichever is more frequent.

The percent recovery of the aqueous LCS should be within the control limits of (80-120%, except for silver and antimony). The percent recovery of the solid LCS must be within control limits given for the standard reference material used. If the results are not within the control limits, the problem is investigated, corrected, and the associated samples are re-digested and re-analyzed along with a new LCS and preparation blank.

9.4 Matrix Spike (MS)

At least one matrix spike is prepared and analyzed with each group of samples of similar matrix. The percent recovery of the matrix spike should be within the limits of 75-125%. If it is not, the results of the associated sample data is flagged with an "N" on the deliverable forms unless the sample concentration exceeds the value of spike added by a factor of four or more. In the latter event, the data is reported without qualification.

If the recovery of the matrix spike is outside control limits and the sample result does not exceed four times the concentration of spike added, a post-digestion spike is performed on the same sample that was used for the matrix spike for those elements that do not meet criteria**.** The sample aliquot for the post-digestion spike should be spiked at two times the indigenous level or two times the CRQL whichever is greater.

9.5 Sample Duplicate (SD)

One sample duplicate sample is prepared and analyzed from each group of samples of a similar matrix. The relative percent difference (RPD) between duplicate results should be less than or equal to 20%, when sample values are greater than or equal to five times the CRQL. If the results are less than 5 times the CRQL, a control limit of the CRQL value should be reported on the deliverable form, otherwise the control limit field is left empty. If one result is above five times the CRQL and the other is below, the CRQL criteria is used for control. If both results are less than the MDL, the relative percent difference is not calculated. For soil samples, the CRQL is corrected for sample weight and percent solids. If the criteria are not met, the associated samples are flagged with a "*" on the deliverable forms.

9.6 Serial Dilution

A serial dilution (five-fold dilution) is performed on a sample from each group of samples of a similar matrix. If the analyte concentration in the undiluted sample is sufficiently high (a factor of 50 above the MDL), the serial dilution should agree within 10% of the original

concentration after correction for dilution. If it is not, a chemical or physical interference is suspected and the data for the associated samples is flagged with an "E" on the deliverable forms.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Calibration

Calibrate the instrument with a blank and one calibration standard for each element at the beginning of each analytical sequence using the instrument operating conditions established by the manufacturer of the instrument. Operating instructions for the instrument are described in the instrument manual(s) located in the metals laboratory.

The calibration blank and standard(s) are prepared per the instructions given in Appendix A and analyzed following the procedure given in Section 11.0. Performance check and continuing calibration standards are interspersed between blanks, QC and field samples throughout the analytical run to ensure on-going accuracy and linearity of the calibration. An example analytical sequence is given in Section 11.0

10.2 Initial Calibration Verification (ICV)

Immediately after the instrument has been calibrated, verify the accuracy of the calibration by analysis of a second source ICV standard that has been prepared in the same acid matrix as the calibration standards. The results of the ICV standard should be within the established limits of 90-110%. If it is not, stop the analysis, correct the problem, recalibrate and initiate a new analytical sequence.

10.3 Continuing Calibration Verification (CCV)

To ensure the accuracy of the calibration during the analytical run, analyze a CCV every 10 samples or every 2 hours, whichever is more frequent and at the beginning and end of each analytical run. The concentration of the elements in the CCV should be at or near the mid points of their respective calibration curve and must be different than the concentration of the ICV. The result of the CCV should be within the established limits of 90-110%. If it is not, stop the analysis, correct the problem, recalibrate and initiate a new analytical sequence.

10.4 Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

To verify linearity near the CRQL, analyze a CRI at the beginning of each analytical run but not before the ICV and once per every 20 samples per analytical run. The CRI shall be run for every wavelength used for analysis, except those for Al, Ba, Ca, Fe, Mg, Na, and K. The percent recovery of the CRI should be within 70-130% (50-150% for Sb, Pb, and Tl). If it is not, stop the analysis, correct the problem, recalibrate and initiate a new analytical sequence.

10.5 Initial and Continuing Calibration Blanks (ICB/CCB)

Analyze calibration blanks at each wavelength after every ICV and CCV, at a frequency of 10% or every 2 hours during the analytical sequence, whichever is more frequent. The blanks are also analyzed at the beginning of the run and after the last sample (after the last CCV). The absolute value of the ICB/CCB must be less than the CRQL. If it is not, stop the analysis, correct the problem, recalibrate and initiate a new analytical sequence.

10.6 Support Equipment Calibration

Check the calibration of the auto-pipettes and the top-loading balance on the day of use prior to use. Record the calibration check in the logbook designated for this purpose.

11.0 PROCEDURE

11.1 Water/Aqueous Sample Preparation (HW1) / EPA 200.7

Shake the sample to mix and transfer 100 mL to a labeled digestion vessel. Transfer two additional aliquots of the sample selected as the sample duplicate (SD) and the matrix spike (MS) to a labeled digestion vessel. Add 1mL of matrix spike solution to the matrix spike (MS) aliquot.

Note: A post digestion spike and serial dilution analyses are routinely performed on the same sample that was designated as the matrix spike. For initial analysis, the concentration of the post digestion spike standard is equivalent to 2X the CRQL for all elements. If the matrix spike recovery for any element is outside control limits, the unspiked sample result for that element does not exceed four times the concentration of spike added and if the indigenous level in the unspiked sample for that element is higher than 2X the CRQL, then the post-digestion spike must be repeated with a spike concentration equivalent to 2X the indigenous level in the unspiked sample for those elements.

To prepare the preparation blank (PB), transfer 100 mL of reagent water to a labeled digestion vessel.

To prepare the LCS, transfer 50 mL of the aqueous LCS standard to a labeled digestion vessel and dilute to 100 mL with 50 mL of reagent water.

Add 2 mL of 1:1 HNO_3 and 10mL of 1:1 HCl to each digestion vessel. Cover the digestion vessel(s) with a watch glass and place in a heated block digester maintained at a temperature of 92-95°C. Heat until the sample volume is reduced between 25 and 50 mL. Monitor the digestion to ensure that the sample does not boil and that the digestion vessel does not go dry. After digestion is complete, cool the digestate, adjust the volume to 100 mL with reagent water and allow any insoluble material to settle.

11.2 Soil Sample Preparation (HS2) SW-846 3050B

Weigh (to the nearest 0.01g) a 1.0 to 2.0 g of sample into a tared labeled digestion vessel. Weigh two additional aliquots of the sample selected as the sample duplicate (SD) and

matrix spike (MS) to individual tared, labeled digestion vessel(s). Add 2 mL of matrix spike solution to the matrix spike (MS) aliquot.

Note: A post digestion spike and serial dilution analyses are routinely performed on the same sample that was designated as the matrix spike. For initial analysis, the concentration of the post digestion spike standard is equivalent to 2X the CRQL for all elements. If the matrix spike recovery for any element is outside control limits, the unspiked sample result for that element does not exceed four times the concentration of spike added and if the indigenous level in the unspiked sample for that element is higher than 2X the CRQL, then the post-digestion spike must be repeated with a spike concentration equivalent to 2X the indigenous level in the unspiked sample for those elements.

To prepare the preparation blank (PB), weigh (to the nearest 0.01g) a 1.0 to 2.0 g of reagent water into a labeled digestion vessel.

To prepare the LCS, weigh (to the nearest 0.01g) a 1.0 to 2.0 g of solid LCS standard material into a labeled digestion vessel.

Add 10ml of 1:1 HNO_3 to each digestion vessel. Cover with a watch glass and place in a heated block digester maintained at a temperature of 92-95°C. Heat the sample for 10 minutes without boiling. Allow the sample to cool, and 5mL of concentrated HNO_3. Replace the watch glass and reflux for 30 minutes. Add an additional 5ml of concentrated HNO_3 and reflux. Repeat this step until no brown fumes are given off by the sample indicating that oxidation is complete.

Allow the sample to cool and add 2 mL of reagent water and 3 mL of 30% hydrogen peroxide. Replace the watch glass and return the digestion vessel to the block digester. Warm to initiate the peroxide action and continue to heat until the effervescence subsides. Allow the sample to cool. Continue to add 30%hydrogen peroxide in 1mL aliquots with warming until the effervescence is minimal or until sample appearance remains unchanged. Do not add more than 10mL of 30% hydrogen peroxide. Add 10mL of concentrated HCl to each digestion vessel. Replace the watch glass and return the digestion vessel to the block digester. Heat for 10 minutes. After digestion is complete, cool the digestate, adjust the volume to 100mL with reagent water and allow any insoluble material to settle.

11.3 Standards Preparation

Prepare the mixed calibration standards and on-going performance check standards (ICV, CCV, CRI, ICSA, ICSAB) using the formulation given in Appendix A. Transfer approximately 25 mL of each prepared standard to individual, labeled autosampler tubes.

Prepare each calibration blank by transferring approximately 25 mL of 5% hydrochloric acid/2% nitric acid solution into individual, labeled autosampler tubes.

11.4 ICP-AES Analysis

Set up and profile the instrument using the operating conditions recommended by the manufacturer. Allow sufficient time for the instrument to become thermally stable prior to

calibration.

For each sample, transfer approximately 8mL of digestate to individual autosampler tubes. Prepare the serial dilution by transferring 1mL of parent sample and 4 mL of the 5% hydrochloric acid/2% nitric acid solution to an autosampler tube.

Prepare a post digestion spike by transferring 0.8 mL of the post-spike solution and 7.2 mL of parent sample to an autosampler tube. To prepare the serial dilution, transfer 1 mL of the sample into an autosampler tube and add 4 mL of reagent water.

On the PC connected to the instrument, select the autosampler template and enter the sample Ids in the order of analysis. Place the samples, serial dilution, post-digestion spike, calibration blanks, mixed calibration standards, and performance check standards in the position on the autosampler rack that corresponds to their assigned position in the autosampler template. Place the autosampler rack in the autosampler tray and initiate the software macro to begin analysis.

An example analytical sequence is given below:

Calibration Blank
Calibration Standard #7
Calibration Standard #8
Calibration Standard #4
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
10 Samples*
CCV
CCB
7 Samples*
CRI
ICSA
ICSAB
CCV
CCB

**The number of samples between each CCB/CCV (10) includes preparation blanks, laboratory control samples, matrix spikes, sample duplicates, serial dilutions and the post digestion spike.*

After analysis is complete, review the data against the criteria given in Section 9.0 for Quality Control and Section 10.0 for Calibration and Standardization. Perform corrective action, as needed. Dilute and reanalyze any samples that exceed the linear range and perform post digestion spikes as necessary.

Transfer the data from the network server into the MARRS data processing software program. Route preparation log, analysis run log and associated raw data to the Inorganic Data Review department for secondary data assessment and report generation.

Note: The data processing software is configured to acquire two replicate exposures for all QC and sample analyses and to use the average result of multiple exposures for standardization. The data system determines the concentration using the equations given in Section 12.0.

12.0 CALCULATIONS

Unless otherwise specified, calculations are performed using data analysis software.

12.1 Equation 1: Water Sample Concentration

$$C_{(ug/L)} = \frac{\mu g}{L_{dig}} * \frac{V_{dig}}{V_{samp}}$$

Where:
$\mu g/L_{dig}$ = ICP result including all dilution factors
V_{dig} = final digestate volume in mLs
V_{samp} = sample volume in mLs

12.2 Equation 2: Soil/Sediment Sample Concentration

Where:
$\mu g/L_{dig}$ = ICP result including all dilution factors
V_{dig} = final digestate volume in Liters
g_{samp} = sample weight in grams

13.0 DATA ASSESSMENT, CRITERIA AND CORRECTIVE ACTION

13.1 Primary review is performed and documented by the analyst that performed the digestion or analysis procedure. All data undergoes secondary review by a senior analyst or data review analyst. The review ensures that all data is reviewed against the performance criteria given in Section 9.0 for quality control and Section 10.0 for calibration and standardization. If the results are not within the established limits or criteria, corrective action is performed. If corrective action is not taken or is unsuccessful, the situation is documented and reported in the project narrative. All data that does not meet established criteria is flagged with the

appropriate data qualifier and noted in the project narrative.

14.0 METHOD PERFORMANCE

14.1 Method Detection Limit (MDL) determinations are determined annually on each instrument for each digestion procedure, and non-prepared analyses following the procedure described in 40CFR, Part 136, Appendix B and laboratory SOP LP-LB-009. The determined MDL must be less than one-half the CRQL. The non-digested MDL is used to determine data qualifiers for the results of the non-prepared samples and QC analyses.

14.2 Interelement Corrections

Interelement correction factors are determined quarterly by the analysis of a single element standard for each element at an environmentally representative concentration. For each of those analyses the presence of a positive or negative value whose absolute value for any other element exceeds the CRQL is documented. A “K” factor is determined by dividing the concentration observed of a given element by the concentration of the interfering element and is entered into the software system. Following this, the ICSA solution is analyzed and the process is repeated for any elements not present in the solution that exhibit a positive or negative value. When the procedure is complete, all “K” values are considered final if the ICSA analysis meets the acceptance criteria for the ICSA analysis.

14.3 Linear Range Analysis

Linear range analysis is determined quarterly by the analysis of a multi-component or single element standard for each element. The highest concentration recovered within 5% of its true value determines the linear range for the instrument.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 The following waste streams are produced when this method is carried out.

- Acidic Waste – Satellite Container: 5 Gallon Polyethylene Container

Transfer the waste stream to the designated satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-0010 *Hazardous Waste.*

16.0 REVISION HISTORY

16.1 Title Page: Updated to current management team.

16.2 Section 5.0: Updated safety information.

16.3 Section 6.0: Updated instrument descriptions.

16.4 Section 7.0: Updated reagent information.

16.5 Section 15.0: Updated pollution prevention and waste management to reflect current practice and added waste stream information.

16.6 Section 18.0: Added Table 2.

17.0 REFERENCES

16.1 CLP SOW ILM05.3 For Inorganic Analyses, Multi-Media, Multi-Concentration, USEPA Office of Solid Waste and Emergency Response, February, 2004.

18.0 TABLES, DIAGRAMS & FLOWCHARTS

18.1 Table 1: TCL List, CRQLs and Analyte Wavelengths Used by Instrument
18.2 Table 2: Primary Materials Used
18.3 Appendix A: Standard Preparation Tables
18.4 Appendix B: List of Definitions

Table 1: Target Analyte List (TCL), CRQLs and Analyte Wavelengths Used by Instrument

Analyte	CAS No.	CRQL		Analyte Wavelengths Used (nm) by Instrument		
		Water (ug/L)	Soil (mg/kg)	TJA Trace ICP4	TJA Trace ICP5	TJA Trace ICP6
Aluminum	7429-90-5	200	20	308.215	237.313	308.215
Antimony	7440-36-0	60	6	206.838	206.838	206.838
Arsenic	7440-38-2	10	1	189.042	189.042	189.042
Barium	7440-39-3	200	20	493.409	493.409	493.409
Beryllium	7440-41-7	5	0.5	313.042	313.042	313.042
Cadmium	7440-43-9	5	0.5	226.502	226.501	226.502
Calcium	7440-70-2	5000	500	317.933	317.933	317.933
Chromium	7440-47-3	10	1	267.716	267.716	267.716
Cobalt	7440-48-4	50	5	228.616	228.611	228.616
Copper	7440-50-8	25	2.5	324.754	324.754	324.753
Iron	7439-89-6	100	10	271.441	271.441	271.441
Lead	7439-92-1	10	1	220.353	220.353	220.353
Magnesium	7439-95-4	5000	500	279.078	279.079	279.078
Manganese	7439-96-5	15	1.5	257.610	294.920	257.610
Nickel	7440-02-0	40	4	231.604	231.601	202.030
Potassium	7440-09-7	5000	500	766.491	776.491	766.491
Selenium	7782-49-2	35	3.5	196.026	196.026	196.026
Silver	7440-22-4	10	1	328.068	328.068	328.068
Sodium	7440-23-5	5000	500	330.232	330.232	330.232
Thallium	7440-28-0	25	2.5	190.864	421.552	190.864
Vanadium	7440-62-2	50	5	292.402	292.402	292.402
Zinc	7440-66-6	60	6	231.856	213.851	206.200

Table 2: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: Standard Preparation Tables

Unless otherwise specified all standards are prepared with a 5% HCl / 2% HNO3 solution.

Mixed Element Calibration Standards

Calibration Standard #7

Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
500ppm Calcium	200	2000	50000
500ppm Potassium			50000
500ppm Sodium			50000
500ppm Magnesium			50000
500ppm Aluminum			50000
500ppm Iron			50000

Calibration Standard #8

Single Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
1000ppm Arsenic	1.0	2000	500
1000ppm Selenium	1.0	2000	500
1000ppm Thallium	1.0	2000	500
1000ppm Antimony	1.0	2000	500
1000ppm Lead	2.0	2000	1000
1000ppm Tin	2.0	2000	1000
1000ppm Strontium	2.0	2000	1000
1000ppm Titanium	2.0	2000	1000

Calibration Standard #4

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
Silver	1.0	2000	500
Phosphorus	2.0		1000
Silicon	10		5000
Boron	20 X-AQU-5		1000
Barium			1000
Beryllium			500
Cadmium			500
Cobalt			1000
Chromium			1000
Copper			1000
Manganese			1000
Molybdenum			1000
Nickel			1000
Vanadium			1000
Zinc			1000

CRI Standards

CRI Working Standard Solution

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
CLP-AES-CRQL Stock	20	2000	See below
1000ppm Sn	0.08	2000	40
1000ppm B	0.4	2000	200
1000ppm Mo	0.04	2000	20
1000ppm Sr	0.08	2000	40
1000ppm P	1.0	2000	500
1000ppm Ti	0.08	2000	40
1000ppm Si	0.4	2000	200

CLP-AES-CRQL: Stock Standard Solution

Stock Standard Used – Element	Initial Standard Concentration (ug/mL)	Final Standard Concentration (ug/L)
Ca Mg K Na	500	5000
Al Ba	20	200
Fe	10	100
Sb Zn	6	60
Co V	5	50
Ni	4	40
Se	3.5	35
Cu Tl	2.5	25
As Mn	1.5	15
Cr Pb Ag	1	10
Be Cd	0.5	5

Interference Check (ICS) Solutions

ICSA Working Standard Solution

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
CLPP-ICS-A Stock	100	2000	See below

CLPP-ICS-A Stock Standard Solution

Stock Standard Used – Element	Initial Standard Concentration (mg/L)	Final Standard Concentration (mg/L)
Al Ca Mg	5000	250
Fe	2000	100

ICSAB Working Standard Solution

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
CLPP-ICS-A Stock	100	2000	See above
CLPP-ICS-B4 Stock	20	2000	See below
1000ppm B	3	2000	1500
1000ppm Sn	3	2000	1500
1000ppm Mo	2	2000	1000
1000ppm Si	2	2000	1000
1000ppm P	1	2000	500
1000ppm Ti	1	2000	500
1000ppm Sr	0.5	2000	250

CLPP-ICS-B4: Stock Standard Solution

Stock Standard Used – Element	Initial Standard Concentration (mg/L)	Final Standard Concentration (ug/L)
Cd Ni Zn	100	1000
Sb	60	600
Ba Be Co Cr Cu Mn V	50	500
Ag	20	200
As Tl	10	100
Pb Se	5	50

Initial Calibration Verification (ICV)

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (ug/L)
100ppm Aluminum	10 AT-2	1000	26,000
100ppm Lead			1000
50ppm Barium			500
50ppm Beryllium			500
50ppm Boron			500
50ppm Cadmium			500
50ppm Chromium			500
50ppm Cobalt			500
50ppm Iron			25,500
50ppm Manganese			500
50ppm Nickel			500
50ppm Silver			500
50ppm Strontium			500
50ppm Vanadium			500
50ppm Zinc			500
50ppm Copper			500
500ppm Aluminum	50 AT-3		See Above
500ppm Calcium			25,000
500ppm Iron			See Above
500ppm Magnesium			25,000
500ppm Potassium			25,000
500ppm Sodium			25,000
1000ppm Molybdenum	0.5		500
1000ppm Antimony	0.25		250
1000ppm Thallium	0.25		250
1000ppm Selenium	0.25		250
1000ppm Arsenic	0.25		250
1000ppm Tin	0.25		250
1000ppm Titanium	0.5		500
1000ppm Phosphorus	0.5		500
1000ppm Silicon	0.25		250

Continuing Calibration Verification (CCV)

CCV Working Standard Solution

Single or Multi-Element Stock Standard	Stock Standard Concentration (ppm)	Volume Stock (mL)	Volume Prepared (mL)	Final Concentration (ug/L)
Silver	Cal Standard #4	200	1000	100
Phosphorus				200
Silicon				1000
Boron				700
Barium				200
Beryllium				100
Cadmium				100
Cobalt				200
Chromium				200
Copper				200
Manganese				200
Molybdenum				200
Nickel				200
Vanadium				200
Zinc				200
Calcium	Cal Standard #7	600		30,200
Potassium				30,200
Sodium				30,200
Magnesium				30,200
Aluminum				30,200
Iron				30,200
Arsenic	Cal Standard #8	200		100
Selenium				100
Thallium				100
Antimony				300
Lead				400
Tin				200
Titanium				400
Strontium				300
Boron	1000 ppm	0.5		See Above

Matrix Spike Solution

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)
10000 ppm Al	10	500	200
1000 ppm Sb	5	500	10
1000 ppm As	2	500	4
1000 ppm Ba	100	500	200
1000 ppm Be	2.5	500	5
1000 ppm Cd	2.5	500	5
1000 ppm Cr	10	500	20
1000 ppm Co	25	500	50
1000 ppm Cu	12.5	500	25
10000 ppm Fe	5	500	100
1000 ppm Pb	1	500	2
1000 ppm Mn	25	500	50
1000 ppm Ni	25	500	50
1000 ppm Se	2.5	500	5
1000 ppm Tl	2.5	500	5
1000 ppm V	25	500	50
1000 ppm Zn	25	500	50
1000 ppm B	25	500	50
1000 ppm Mo	25	500	50
1000 ppm Sn	25	500	50
1000 ppm Si	25	500	50
1000 ppm P	25	500	50
1000 ppm Ti	25	500	50
1000 ppm Ag	2.5	500	5
1000 ppm Sr	25	500	50

Aqueous LCS Solution

Single or Multi-Element Stock Standard	Volume Used (mL)	Final Volume (mL)	Final Concentration (mg/L)
**AT - 2	40	2000	See below
***AT - 3	400	2000	See below
1000ppm Sb	8	2000	4
1000ppm As	4	2000	2
1000ppm Sn	4	2000	2
1000ppm Mo	4	2000	2
1000ppm Se	2	2000	1
1000ppm Tl	2	2000	1
1000ppm Sr	4	2000	2
1000ppm Si	4	2000	2
1000ppm P	4	2000	2
1000ppm Ti	4	2000	2
****LCSWF	20	2000	See below

**** AT - 2**

Stock Standard Used – Element	Initial Concentration (mg/L)	Final Standard Concentration (mg/L)
Al and Pb	100	2
Ba, Be, B, Cd, Cr, Co, Fe Mn, Ni, Ag, Sr, V and Zn	50	1

***** AT - 3**

Stock Standard Used – Element	Initial Standard Concentration (mg/L)	Final Standard Concentration (mg/L)
Al, Ca, Fe, Mg, K and Na	500	100

****** **LCSWF**

Stock Standard Used – Element	Initial Standard Concentration (mg/L)	Final Standard Concentration (mg/L)
As and Tl	10	0.1
Cd and Se	5	0.05
Pb	3	0.03

Appendix B: List of Definitions

ANALYTE - The element or ion an analysis seeks to determine; the element of interest.

ANALYTICAL SEQUENCE - The actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final CCV or CCB.

BACKGROUND CORRECTION - A technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

BATCH - A group of samples prepared at the same time in the same location using the same method.

BLANK - An analytical sample designed to assess specific sources of contamination.

CALIBRATION - The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards.

CALIBRATION BLANK - A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation.

CALIBRATION STANDARDS - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

CONTINUING CALIBRATION VERIFICATION (CCV) - A single parameter or multiparameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples.

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) - Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) CHECK STANDARD (CRI) - A single parameter or multi-parameter standard solution prepared at the CRQL and used to verify the instrument calibration at low levels.

CONTROL LIMITS - A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

DISSOLVED METALS - Analyte elements in a water/aqueous sample which will pass through a 0.45 micrometer (µm) filter.

DRY WEIGHT - The weight of a sample based on percent solids. The weight after drying in an oven.

DUPLICATE - A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

HOLDING TIME - The elapsed time expressed in days from the date of receipt of the sample by the Contractor until the date of its analysis.

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY (ICP-AES) – A technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma.

INITIAL CALIBRATION - Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

INITIAL CALIBRATION VERIFICATION (ICV) - Solution(s) prepared from stock standard solutions, metals or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration.
.
INTERFERENCE CHECK SAMPLE - A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

INTERFERENTS - Substances which affect the analysis for the element of interest.

LABORATORY CONTROL SAMPLE (LCS) - A control sample of known composition.

LINEAR RANGE, LINEAR DYNAMIC RANGE - The concentration range over which the instrument response remains linear.

MATRIX - The predominant material of which the sample to be analyzed is composed.

MATRIX EFFECT - In general, the effect of particular matrix constituents.

MATRIX SPIKE - Aliquot of a sample (water/aqueous or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

METHOD DETECTION LIMIT (MDL) - The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where “s” is the standard deviation of the 7 replicates.

PERCENT DIFFERENCE (%D) - The difference between the two values divided by one of the values.

PERCENT SOLIDS (%S) - The proportion of solid in a soil sample determined by drying an aliquot of the sample.

PREPARATION BLANK - An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

RELATIVE PERCENT DIFFERENCE (RPD) - The relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

SAMPLE - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SENSITIVITY - The slope of the analytical curve (i.e., functional relationship between instrument response and concentration).

SERIAL DILUTION - The dilution of a sample by a factor of five. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

STOCK SOLUTION - A standard solution which can be diluted to derive other standards.

TARGET ANALYTE LIST (TAL) - A list of Inorganic Analytes (metals and cyanide) as designated in Exhibit C.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - The date on which a sample is received at the Contractor’s facility, as recorded on the shipper’s delivery receipt and Sample Traffic Report/Chain of Custody Record.

STANDARD OPERATING PROCEDURE
STL BURLINGTON

CLP SOW ILM05.3 / Mercury Cold Vapor Analysis

Applicable Matrices: Waters, Soil, Sediment and Sludge

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the analysis of total mercury in water, sediment, sludge and soil samples taken from hazardous waste sites using a cold vapor technique with atomic absorption (AA).

1.2 The normal range of determination may vary by instrument conditions and/or increasing/decreasing sample size. For aqueous samples, the contract required detection limit (CRQL) is 0.2ug/L, and for soils, the CRQL is 0.1mg/kg.

2.0 SUMMARY OF METHOD

2.1 Water Samples

A measured volume of sample is acid digested with potassium permanganate and potassium persulfate for two hours in a block digester maintained at a temperature of 95°C. After digestion, hydroxylamine hydrochloride is added to each digestate in order to reduce excess permanganate. The digestate is placed on a closed-system mercury autoanalyzer and stannous chloride is added to each sample. The elemental mercury released is measured spectrophotometrically at a wavelength of 253.7 nm. The concentration is calculated from the response of the sample absorbance applied against the calibration curve.

2.2 Soil/Sediment Samples

A weighed portion of solid sample is acid digested for 2 minutes at a temperature of 95°C then digested with potassium permanganate and potassium persulfate for 30 minutes at a temperature of 95°C. Hydroxylamine hydrochloride is added to each digestate in order to reduce excess permanganate. The digestate is placed on a closed-system mercury autoanalyzer and stannous chloride is added to each sample. The elemental mercury released is measured spectrophotometrically at a wavelength of 253.7 nm. The concentration is calculated from the response of the sample absorbance applied against the calibration curve.

2.3 This procedure is based on CLP SOW ILM05.3, Exhibit D (Mercury).

3.0 DEFINITIONS

3.1 A list of definitions is given in Appendix B.

4.0 INTERFERENCES

4.1 Water

Some seawaters and wastewaters that are high in chlorides exhibit a positive interference, During the oxidation step chlorides are converted to free chlorine which will also absorb radiation at 253 nanometers (nm). To ensure free chlorine is absent before the mercury is

reduced and swept into the cell use an excess of hydroxylamine HCl reagent (25mL) and additional amounts of permanganate [as much as 25 milliliters (mL)].

The formation of a heavy precipitate, in some wastewaters and effluents, has been reported upon addition of concentrated sulfuric acid. If this is encountered, the sample cannot be analyzed by this procedure.

Possible interference from sulfide is eliminated by the addition of potassium permanganate. Copper has also been reported to interfere; however, per the CLP SOW, copper concentrations as high as 10mg/L have not shown an effect on recovery of mercury from spiked samples.

If total mercury values are to be reported, water samples that contain solids must be blended and then mixed during field sampling.

4.2 Soil/Sediment

The same types of interferences noted for water samples are possible with sediments.

Samples that contain high concentrations of oxidizable organic materials, (high COD values) may not be completely oxidized by this procedure. If this occurs, the recovery of organic mercury will be low. To eliminate the problem, reduce the weight of the original samples or increase the amount of potassium persulfate (and stannous chloride) used in the digestion.

5.0 SAFETY

5.1. Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added. Protective clothing such as a lab coat, safety glasses and latex gloves must be worn while performing this procedure.

5.3. Primary Materials Used

Table 1, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure**.** A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 Mercury Auto-Analyzer; Leeman Labs PS 200; Leeman Labs PS 200II, Leeman Labs Hydra AA.

6.2 Block digester; Environmental Express

6.3 Polyethylene Digestion Vessels; 100 mL; Environmental Express

6.4 Volumetric Pipets

6.5 Top Loading Balance

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent Water, ASTM Type II

Sulfuric Acid, concentrated - Reagent grade; JT Baker or equivalent

Nitric Acid, concentrated - Reagent grade; JT Baker or equivalent

Stannous Chloride; Reagent grade; JT Baker or equivalent

Hydroxylamine Hydrochloride; Reagent grade; JT Baker or equivalent

Potassium Permanganate; Reagent grade; JT Baker or equivalent

Potassium Persulfate; Reagent grade; JT Baker or equivalent

Stannous Chloride: Add 100g of stannous chloride to 1L of 10% hydrochloric acid.

Hydroxylamine Hydrochloride: Dissolve 240grams of Hydroxylamine Hydrochloride in 2L of reagent water.

Potassium Permanganate ($KMNO_4$): 5% solution w/v: Dissolve 100 grams of Potassium Permanganate in 2L of reagent water.

Potassium Persulfate – 5% solution w/v: Dissolve 100 grams of Potassium Persulfate in 2L of reagent water.

7.2 Standards

Stock standard solutions are purchased from commercial vendors. Intermediate and working standard solutions are prepared from stock standards by diluting volumes of stock standards with acidified reagent water in volumetric flasks to obtain the appropriate concentration. Expiration dates of prepared solutions are assigned according to expiration

date of the parent standard. Standard solutions are prepared as needed or on the expiration date, whichever occurs first.

Mercury Stock Standard Solution (1000mg/L): Purchased from Spex.

Mercury Intermediate Standard (10,000ug/L): Add 1mL of the 1000mg/L stock standard solution and 0.15mL of concentrated HNO_3 to a 100mL volumetric flask that contains approximately 80mL reagent water. Adjust to volume with reagent water.

Mercury Working Standard (100ug/L): Add 10mL of the intermediate standard and 1.5mL of concentrated HNO_3 to a 1000mL volumetric flask that contains approximately 800mL reagent water. Adjust to volume with reagent water. Secondary dilutions of the working standard are used for the calibration curve, continuing calibration verification (CCV) standards, and the Contract Required Quantitation Limit (CRQL) Check Standard (CRI).

ICV Stock Standard Solution (1000mg/L): Purchased from Inorganic Ventures.

ICV Intermediate Standard Solution (10,000ug/L): Add 1mL of the 1000mg/L ICV stock standard solution and 0.15mL of concentrated HNO_3 to a 100mL volumetric flask that contains approximately 80mL reagent water. Adjust to volume with reagent water.

ICV Working Standard Solution (30ppb): Add 1.5mL of the ICV intermediate standard and 0.75mL of concentrated HNO_3 into a 500mL volumetric flask that contains approximately 300mL reagent water. Adjust to volume with reagent water.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

8.1 All samples should be collected in glass or polyethylene containers and iced or refrigerated to 4°C (±2°C) from the time of collection until digestion. Water samples must be preserved nitric acid to pH less than 2 immediately after collection. If dissolved metals are to be determined, the samples must be filtered through a 0.45 micrometer (µm) pore diameter membrane filter at the time of collection or as soon as possible. Preserve the filtrate with nitric acid to pH less than 2 immediately after filtration.

8.2 The maximum holding time for mercury is 26 days from Validated Time of Sample Receipt (VTSR).

8.4 Samples are stored protected from light and refrigerated at 4^oC ($\pm$ 2^oC) from the time of receipt until 60 days after delivery of a reconciled, complete sample data package to the client. After 60 days, the samples are disposed of in a manner that complies with all applicable regulations.

9.0 QUALITY CONTROL

9.1 Preparation Blank (PB)

A PB is prepared and analyzed with each digestion batch or every sample delivery group, whichever is more frequent. The concentration of mercury in the blank must be less or

equal to the CRQL. If this criterion is not met, the lowest concentration of mercury in the samples must be greater than or equal to 10 times the concentration in the blank. Otherwise, the blank and associated samples are re-digested and re-analyzed. If the concentration of the blank is below the negative CRQL, all samples associated with the blank that have mercury concentrations below 10 times the CRQL are re-digested and re-analyzed.

9.2 Laboratory Control Sample (LCS)

A solid laboratory control sample is prepared and analyzed with each soil digestion batch. The percent recovery of the LCS must be within the control limits specified for the standard reference material (usually 85-115%). If the results are not within the control limits, the problem should be investigated, corrected, and the associated samples must be re-digested and re-analyzed along with the LCS and preparation blank.

9.3 Matrix Spike (MS)

At least one matrix spike is prepared and analyzed with each digestion batch or every sample delivery group whichever is more frequent. The MS provides information about the effect of the sample matrix on the digestion or analysis process and is analyzed in the same manner as the unspiked sample. The percent recovery of the MS should be within the limits of 75-125%. If it is not, the results of associated sample data are flagged with an “N” on the deliverable forms unless the sample concentration exceeds the value of spike added by a factor of four or more. In the latter event, the data is reported without qualification.

9.4 Sample Duplicate (SD)

One duplicate sample is prepared and analyzed from each group of samples of a similar matrix. The relative percent difference between duplicate sample results greater than or equal to the CRQL should be less than or equal to 20%. If the results are less than 5 times the CRQL, a control limit of the CRQL is reported on the deliverable forms, otherwise the control limit field is left empty. If one result is above five times the CRQL and the other is below, the CRQL criteria is used for control. If both results are less than the MDL, the relative percent difference is not calculated. For soil samples, the CRQL is corrected for sample weight and percent solids. If the criteria are not met, the associated samples are flagged with a “*” on the deliverable forms.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Initial Calibration

The mercury autoanalyzers are calibrated with five calibration standards and a blank at the beginning of each analytical sequence using the instrument operating conditions established by the manufacturer of the instrument. Operating instructions for the instrument are described in the instrument manual(s) located in the laboratory.

The calibration standards are prepared daily by making successive dilutions of the working standard (100ug/L). The final concentration of the prepared calibration standards is given in

the following tables:

Table I - Water

Level	Working Standard(100ug/L) mL	Final Volume mL	Final Concentration* ug/L
Blank	0	50	0
Level 1	0.1	50	0.2
Level 2	0.25	50	0.5
Level 3	0.5	50	1
Level 4	2.5	50	5
Level 5	5.0	50	10

Table II - Soil

Level	Working Standard (100 ug/L) mL	Final Volume mL	Final Concentration* ug/L
Blank	0	5.0	0
Level 1	0.1	5.0	0.2
Level 2	0.25	5.0	0.5
Level 3	0.5	5.0	1
Level 4	2.5	5.0	5
Level 5	5.0	5.0	10

**The final concentration is achieved after digestion.*

The calibration standards are digested and analyzed following the procedures given in Section 11.0. After analysis, the data system prepares a standard curve by plotting the instrument response of the calibration standards against the true value concentration and using linear regression calculates the correlation coefficient. The correlation coefficient must be greater than or equal to 0.995 and the results for each calibration level except the level equivalent to the CRQL should be within 5% of the true value. If these criteria are not met, the instrument is recalibrated.

10.2 Initial Calibration Verification (ICV)

Immediately after the instrument is calibrated, the accuracy of calibration is verified with analysis of the ICV standard. The ICV standard solution is prepared following the procedure given in Section 7.2 and digested and analyzed with the samples following the procedures given in Section 11.0. The percent recovery of the ICV must be within 80-120%. If it is not, the analysis is stopped the instrument is recalibrated and a new analytical sequence is initiated.

10.3 Continuing Calibration Verification (CCV)

To ensure the accuracy of the calibration during the analysis run, a CCV standard is analyzed at the beginning and end of each analytical run, every 10 samples or every 2 hours, whichever is more frequent. The concentration of the CCV standard should be at or near the mid point of the calibration curve and must be different than the concentration of the ICV. The CCV standard is prepared, digested and analyzed with the samples following

the procedure given in Section 11.0. The percent recovery of the CCV must be within 80-120%. If it is not, the analysis is stopped, the problem is corrected, the instrument is recalibrated and verified and all samples analyzed since the last compliant CCV are reanalyzed.

10.4 CRQL Check Standard (CRI)

To verify linearity near the CRQL, a CRI is analyzed at the beginning of each analytical run but not before the ICV and once per every 20 samples per analytical run. The CRI analysis is immediately followed by analysis of a CCV/CCB pair. The CRI standard is the same (0.2ug/L) solution used as the second level of the calibration curve. The CRI is prepared, digested and analyzed with the samples following the procedure given in Section 11.0. The percent recovery of the CRI should be within 70-130%. If it is not, the analysis is stopped, the problem is corrected the instrument is recalibrated and a new analytical sequence is initiated.

10.5 Initial and Continuing Calibration Blanks (ICB/CCB)

Calibration blanks are analyzed at each wavelength after every ICV and CCV, at a frequency of 10% or every 2 hours during the analytical sequence, whichever is more frequent. The blanks are also analyzed at the beginning of the run and after the last sample (after the last CCV). The ICB/CCB are prepared, digested and analyzed with the samples following the procedure given in Section 11.0. The absolute value of the ICB/CCB must be less than the CRQL. If it is not, the analysis is stopped, the problem is corrected, the instrument is recalibrated and verified and all samples analyzed since the last compliant ICB/CCB are reanalyzed.

10.6 Support Equipment Calibration

Check the calibration of the auto-pipettes and the top-loading balance on the day of use prior to use. Record the calibration check in the logbook designated for this purpose.

11.0 PROCEDURE

11.1 Water Sample and Standard Preparation (Manual)

For each sample, sample duplicate and sample selected as the matrix spike transfer a 50mL aliquot of sample to a labeled digestion vessel. Add 0.5mL of the working standard solution (100ug/L) to the sample selected as the matrix spike.

To prepare the preparation blank, LCS, and calibration blanks, add 50mL of reagent water to a labeled digestion vessel(s). Add 0.5mL of the working standard solution (100ug/L) to the LCS.

Prepare the calibration standards by diluting a volume of the working standard solution (100ppb) in reagent water to achieve the concentrations given in Table I. Transfer the prepared standards to labeled digestion vessel(s).

To prepare the ICV, transfer 5.0mL of the ICV working standard solution (30ppb) to a labeled digestion vessel and adjust the volume to 50mL with reagent water.

To prepare the CCV, transfer 2.5mL of the working standard solution (100ppb) to a labeled digestion vessel and adjust the volume to 50mL with reagent water.

To prepare the CRI, transfer 0.1mL of the working standard solution (100ppb) to a labeled digestion vessel and adjust the volume to 50mL with reagent water.

To each jar, add 2.5mL of concentrated sulfuric acid, 1.25mL of concentrated nitric acid, and 7.5mL of potassium permanganate solution. Allow the bottles to stand for 15 minutes. During this time, the solution should turn and remain the color purple. If after 15 minutes, the solution does not remain purple, add more potassium permanganate (~1.25mL).

Add 4mL of potassium persulfate to each jar and heat for two hours in a block digester maintained at 95°C. After the time period has elapsed, allow the jar to cool then add 3mL of hydroxylamine hydrochloride to each jar in order to reduce the excess permanganate. Swirl to ensure that any soluble residue dissolves back into solution.

Transfer each solution to individual autoanalyzer tubes in preparation for analysis.

11.2 Solid Sample and Standard Preparation (Manual)

For each sample, sample duplicate and sample selected as the matrix spike measure 0.2g of sample into a labeled digestion vessel and add 5.0mL of reagent water. Add 0.5mL of the mercury working standard solution (100ug/L) to the sample selected as the matrix spike.

To prepare the preparation blank and calibration blank(s), add 5.0mL of reagent water to a labeled digestion vessel(s). To prepare the LCS, measure 0.2g of solid LCS material into a labeled digestion vessel and add 10mL of reagent water.

Prepare the calibration standards by diluting a volume of the working standard solution (100ppb) in reagent water to achieve the concentrations given in Table II. Transfer the prepared standards to labeled digestion vessel(s).

To prepare the ICV, transfer 5.0mL of the ICV working standard solution (30ppb) to a labeled digestion vessel.

To prepare the CCV, transfer 2.5mL of the mercury working standard solution (100ppb) into a labeled digestion vessel and adjust the volume to 10mL with reagent water.

To prepare the CRI, transfer 0.1mL of the working standard solution (100ppb) to a labeled digestion vessel and adjust the volume to 50mL with reagent water.

To each jar, add 2.5mL of concentrated sulfuric acid and 1.25mL of concentrated nitric acid. Heat in a hot block digester maintained at 95 C for 2 minutes. After two minutes, remove from heat and allow the bottles to cool. Add 25mL of reagent water, 7.5mL of potassium permanganate, and 4mL of potassium persulfate and swirl to mix. Heat in a hot block

digester maintained at 95 C for 30 minutes. Remove from heat, allow the jars to cool and add 3mL of hydroxylamine hydrochloride to reduce the excess permanganate. Swirl each jar to ensure that any soluble residue dissolves back into solution. If any sample is still purple, add additional hydroxylamine hydrochloride in 3mL increments until the purple color disappears.

Add 25mL of reagent water to each jar and then transfer the solutions to individual autoanalyzer tubes in preparation for analysis.

11.3 Instrument Set Up & Analysis

Turn on the instrument lamp, gas and pump. Allow 15 minutes for the instrument to warm up. Fill the rinse bath with 10% hydrochloric acid solution. Check all tubing connections and reset the calibration curve. Check the stannous chloride reductant reservoir and fill as necessary. Check and record the 0.2ug/L standard intensity. This value must be >2500. When applicable, record sample intensity and reference intensities and verify against previous day. Perform maintenance to increase intensities if needed.

On the PC connected to the instrument, select the autosampler template and enter the sample Ids in the order of analysis. Place the samples, calibration blanks, calibration standards, and performance check standards in the position on the autosampler rack that corresponds to their assigned position in the autosampler template. Place the autosampler rack in the autosampler tray and initiate the software macro to begin analysis.

Example Analytical Sequence:

Calibration Blank
0.2 Calibration Standard
0.5 Calibration Standard
1.0 Calibration Standard
5.0 Calibration Standard
10.0 Calibration Standard
ICV
ICB
CRI
CCV
CCB
9 Samples*
CCV
CCB
9 Samples*
CRI
CCV
CCB

**The number of samples between each CCB/CCV (10) includes preparation blanks, laboratory control samples, matrix spikes, and sample duplicates.*

After analysis is complete, review the data against the criteria given in Section 9.0 for Quality Control and Section 10.0 for Calibration and Standardization. Perform corrective action, as needed. Dilute and reanalyze any samples that exceed the linear range.

Transfer the data from the network server into the MARRS data processing software program. Route preparation log, analysis run log and associated raw data to the Inorganic Data Review department for secondary data assessment and report generation.

Note: The mercury autoanalyzer acquires the data and determines the concentration in using the equations given in Section 12.0.

11.0 CALCULATIONS

12.1 Equation 1: Water Sample Concentration

$$C_{(ug/L)} = \frac{\mu g}{L_{dig}} * \frac{V_{dig}}{V_{samp}}$$

Where:
$\mu g/L_{dig}$ = Instrument result adjusted for dilution factors
V_{dig} = Final digestate volume
V_{samp} = Sample volume

12.2 Equation 2: Soil Sample Concentration

$$C_{(mg/Kg\ drywt.)} = \frac{\mu g}{L_{dig}} * \frac{V_{dig}}{g_{samp}} * \frac{100}{\%\ solids}$$

Where:
$\mu g/L_{dig}$ = Instrument result adjusted for dilution factors
V_{dig} =Final digestate volume
g_{samp} = Sample weight in grams
% Solids = Percent solids to nearest 0.1%

13.0 DATA ASSESSMENT, CRITERIA AND CORRECTIVE ACTION

13.1 All samples, standards and QC samples are reviewed against the performance criteria given in Section 9.0 for quality control and Section 10.0 for calibration and standardization. If the results do not fall within the established limits or criteria, corrective action should be performed. If corrective action is not taken or unsuccessful, the situation must be documented and reported in the project narrative. Primary review of the data is performed by the analyst(s) that performed the procedure. Secondary review is performed by a senior

analyst or a data review analyst. All data that does not meet established criteria must be flagged with the appropriate data qualifier and noted in the project narrative

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) determination is determined annually for each digestion procedure following the procedure described in 40CFR, Part 136, Appendix B and laboratory SOP LP-LB-009. The determined MDL must be less than one-half the CRQL (0.2ug/L and 0.1mg/kg for water and soil samples, respectively).

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is carried out.

- Acid Waste – Satellite Container: 5 Gallon Polyethelyene Container

Transfer the waste stream to the designated satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-0010 *Hazardous Waste.*

16.0 REVISON HISTORY

16.1 Table 1: changed ug/L to mL in working standard title box.

16.2 Table 2: changed final volume for soil calibration standards to 5.0 mL

16.3 Section 11.2: changed regent water addition from 10.0 mL to 5.0 mL. Changed LCS amount from 0.1g to 0.2g.

17.0 REFERENCES

17.1 CLP SOW ILM05.3 For Inorganic Analyses, Multi-Media, Multi-Concentration, USEPA Office of Solid Waste and Emergency Response, February, 2004.

18.0 TABLES, DIAGRAMS & FLOWCHARTS

18.1 Table 1: Primary Materials Used

18.2 Appendix A: *List of Definitions*

Table 1: Primary Materials Used, Hazards & Exposure Limit

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: List of Definitions

ANALYTE - The element or ion an analysis seeks to determine; the element of interest.

ANALYTICAL SEQUENCE - The actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final CCV or CCB.

BATCH - A group of samples prepared at the same time in the same location using the same method.

BLANK - An analytical sample designed to assess specific sources of contamination.

CALIBRATION - The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards.

CALIBRATION BLANK - A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation.

CALIBRATION STANDARDS - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

CONTINUING CALIBRATION VERIFICATION (CCV) - A single parameter or multiparameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples.

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) - Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) CHECK STANDARD (CRI) - A single parameter or multi-parameter standard solution prepared at the CRQL and used to verify the instrument calibration at low levels.

CONTROL LIMITS - A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

DIGESTION LOG - An official record of the sample preparation (digestion).

DRY WEIGHT - The weight of a sample based on percent solids.

DUPLICATE - A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

HOLDING TIME - The elapsed time expressed in days from the date of receipt of the sample by the Contractor until the date of its analysis. Holding time = (sample analysis date - sample receipt date)

INITIAL CALIBRATION - Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

INITIAL CALIBRATION VERIFICATION (ICV) - Solution(s) prepared from stock standard solutions, metals or salts obtained from a source separate from that utilized to prepare the calibration standards.

INTERFERENTS - Substances which affect the analysis for the element of interest.

LABORATORY CONTROL SAMPLE (LCS) - A control sample of known composition. Laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the USEPA samples received.

LINEAR RANGE, LINEAR DYNAMIC RANGE - The concentration range over which the instrument response remains linear.

MATRIX - The predominant material of which the sample to be analyzed is composed. For the purpose of this SOW, a sample matrix is either water/ aqueous or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX EFFECT - In general, the effect of particular matrix constituents.

MATRIX SPIKE - Aliquot of a sample (water/aqueous or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

METHOD DETECTION LIMIT (MDL) - The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where “s” is the standard deviation of the 7 replicates.

PERCENT DIFFERENCE (%D) - As used in this SOW and elsewhere to compare two values. The difference between the two values divided by one of the values.

PERCENT SOLIDS (%S) - The proportion of solid in a soil sample determined by drying an aliquot of the sample.

PREPARATION BLANK - An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in the SOW and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

SAMPLE - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SENSITIVITY - The slope of the analytical curve (i.e., functional relationship between instrument response and concentration).

STOCK SOLUTION - A standard solution which can be diluted to derive other standards.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report/Chain of Custody Record.

ARCADIS BBL

Attachment 1-3

STL SW-846 Organic SOPs

STANDARD OPERATING PROCEDURE
VOLATILE ORGANIC COMPOUNDS BY GC/MS
SW-846 8260B
Applicable Matrices: Non-Potable Water and Solid and Chemical Materials
Standard Compound List and Reporting Limits: See Table 1

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the GC/MS procedure for the analysis of volatile organic compounds in ground and surface water, waste solvents, oily wastes, soils and sediments using a chromatographic column with a temperature program to separate the desorbed purgeables followed by mass spectral detection. This SOP is applicable to the analytical procedure only. The techniques by which compounds may be introduced into the GC/MS system are described in the following SOPs:

LM-MV-5030	Purge and Trap of Aqueous Samples
LM-MV-5035	Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples.

1.2 The analytes that can be determined by this procedure and their associated Reporting Limits (RL) are listed in Table 1, Section 18.

2.0 SUMMARY OF METHOD

2.1 Basic Principles - The analytes are introduced into the GC/MS by purge-and-trap techniques (Method 5030 or Method 5035). Upon desorption from the trap, the volatile compounds are introduced directly to a wide-bore capillary column. A temperature program is used to separate the purgeables. The eluted analytes pass through a jet separator and are carried on the gas stream into the ion source of a mass spectrometer. The ionized molecules are focused and separated according to their mass/charge (m/z) ratio by the quadrupole analyzer. The signal is amplified by an electron multiplier and interpreted by the mass spectrometer data system to produce a total ion chromatogram and mass spectra for every data point on the chromatogram.

2.2 General Method - The mass spectrometer is calibrated to recognize m/z values in the range of 35-300 amu. Reference spectra and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for samples. Analytes are quantified using internal standard calibration. The concentration of each identified component is measured by relating the MS response of the quantification ion produced by that compound to the MS response of the quantification ion produced by a compound that is used as an internal standard. The performance of the mass spectrometer is verified by the injection of 4-Bromofluorobenzene (BFB). Next, the instrument must demonstrate acceptable chemical calibration and linearity by the analysis of five concentrations of a standard mix containing the analytes of interest, as well as the surrogates and internal standards. Before any samples are analyzed, a method blank must be analyzed to demonstrate that the instrument is free from contamination, and that surrogate recovery criteria are met. All analyses must occur within 12 hours of the injection of the passing BFB. Another analytical sequence may be started by the analysis of a passing BFB MS tune followed by a continuing calibration standard.

2.3 This procedure is based on Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (Method 8260B), Revision 2, December 1996, USEPA SW-846 Methods for Evaluating Solid Waste, Update III.

3.0 DEFINITIONS

A list of terms and definitions is given in Appendix C.

4.0 INTERFERENCES

4.1 During analysis, major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of Teflon tubing, Teflon thread sealants, or flow controllers with rubber components in the purging device should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of laboratory reagent blanks provide information about the presence of contaminants. Subtracting blank values from sample results is not permitted.

4.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing relatively high concentrations of volatile organic compounds. The auto-sampler utilizes a single purge vessel that is automatically rinsed between analyses. After analysis of a sample containing high concentrations of volatile organic compounds, one or more laboratory reagent blanks may be analyzed to check for carry-over.

4.3 Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride; otherwise, random background levels will result. Since methylene chloride will permeate Teflon tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory worker's clothing should be cleaned frequently since clothing previously exposed to methylene chloride fumes during common extraction procedures can contribute to sample contamination. Extraction laboratory personnel should not enter the volatile analytical laboratory.

4.4 Traces of ketones, methylene chloride, and some other organic solvents can be present even in the highest purity methanol. This is another potential source of contamination, and should be assessed before standards are prepared in the methanol.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichlorethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride.

5.3 Primary Materials Used

Table 3, Section 18 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **Note: The table does not include all materials used in the procedure. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used can be found in Section 7. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

- Methanol

6.0 EQUIPMENT AND SUPPLIES

6.1 Containers

- Sample Storage Containers: 40 mL screw cap vials equipped with Teflon faced silicone septum, certified clean, known volume of 44 mL (also see Method 5035).
- Standard Storage Containers: 1-5 mL Mininert vials with Teflon lined screw caps

6.2 Computer Hardware/Software: GCMS Acquisition Platform - Hewlett-Packard ChemStations. Data Processing - Hewlett-Packard 9000-series computers, an HP9000 D250 (Chemsvr4) and an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5.

6.3 Instrumentation

- VOA Autosampler: Tekmar ALS 2050, EST Archon, or equivalent
- Purge & Trap: Tekmar LSC 2000, EST Encon; VOCARB 3000 trap or equivalent
- Gas Chromatograph: Hewlett-Packard 5890 Series II and 6890
- Mass Spectrometer: Hewlett-Packard 5971 MSD, Hewlett-Packard 5973 MSD
- Primary Column: Fused silica capillary column, J&W DB624 75 m x 0.53 mm x 3.0 um or equivalent

6.4 Syringes: 250 μL - 10 mL gas tight hypodermic syringes with Luer-Lok tip, Micro syringe 10 - 100 μL

7.0 REAGENTS AND STANDARDS

7.1 Trap Packing Materials - VOCARB 3000 or equivalent traps may be used, following the manufacturer's instructions.

7.2 Reagents

- Methanol - Purge and Trap Grade, demonstrated to be free of analytes.
- Reagent water - Deionized water is filtered using a Milli Q plus ™ filtration system and then boiled for one hour, and purged with helium for a minimum of fifteen minutes. The water is stored in clean, narrow-mouth bottles with Teflon lined septa and screw caps.
- Hydrochloric acid (1:1) - Measured volumes of conc. HCl are carefully added to an equal volume of reagent water.
- Sodium Bisulfate ($NaHSO_4$) Solution - 20% wt/v. Preservative for soil samples (5035).

7.3 Standards

7.3.1. Stock Standard Solutions - These solutions are purchased as certified solutions or prepared from pure standard materials. Commercial standards arrive ampulized in concentrations ranging from 1-5 mg/mL. Preparation of Working Standards from the stock standards is outlined in Appendix A.

7.3.2. Preparation of Calibration Standards

Prepare the five-point calibration curve for the waters and soils using the Working Standards prepared in Appendix A. The volatile water curve is prepared in 44 mL vials; all standards are spiked directly through the septum of the 44 mL vial. Prepare the routine level water and medium level soil curve as follows:

ROUTINE LEVEL WATER AND MEDIUM LEVEL SOIL CALIBRATION CURVE					
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**
8260 Calibration Standard - Mixed	2.2 μL	8.8μL	22 μL	44 μL	88 μL
8260 Calibration Gas - 100 mg/L	2.2 μL	8.8μL	22 μL	44 μL	88 μL
8260 Calibration Added - 100 mg/L	2.2 μL	8.8μL	22 μL	44 μL	88 μL
Internal Standard - 50 mg/L	44 μL	44 μL	44 μL	44 μL	44 μL
Final Volume	44 mL	44 mL	44 mL	44 mL	44 mL
FINAL CONCENTRATION in μg/L					
All analytes except as listed below:	5	20	50	100	200
1,4-Dioxane	250	1000	2500	5000	10000
Isobutyl alcohol	250	1000	2500	5000	10000
Propionitrile	20	80	200	400	800
Tetrahydrofuran	50	200	500	1000	2000
Internal Standards	50	50	50	50	50

As chloroethane often follows a quadratic, rather than linear, pattern, a sixth point may be prepared and analyzed for the calibration gases for this purpose. Even though all the gases will be contained in that calibration point, it is only anticipated that the chloroethane value will be used.

Prepare the low-level soil curve in 44 mL vials containing 5 mL of VOA free water as follows:

LOW LEVEL SOIL CURVE					
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**
8260 Calibration Standard - Mixed	2.2 µL	1.0µL	2.5 µL	5.0 µL	10 µL
8260 Calibration Gas - 100 mg/L	2.2 µL	1.0µL	2.5 µL	5.0 µL	10 µL
8260 Calibration Added - 100 mg/L	2.2 µL	1.0µL	2.5 µL	5.0 µL	10 µL
Internal Standard - 50 mg/L	44 µL	5 µL	5 µL	5 µL	5 µL
Final Volume	44 mL then transfer 5 mL to 44 mL vial	5 mL	5 mL	5 mL	5 mL
FINAL CONCENTRATION in µg/L					
All analytes except as listed below:	5	20	50	100	200
1,4-Dioxane	250	1000	2500	5000	10000
Isobutyl alcohol	250	1000	2500	5000	10000
Propionitrile	20	80	200	400	800
Tetrahydrofuran	50	200	500	1000	2000
Internal Standards	50	50	50	50	50

Prepare the five-point calibration curve for Low Level Waters as follows:

LOW LEVEL WATER CALIBRATION CURVE					
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**
8260 Calibration Standard Low - Mixed	1.8 µL	8.8µL	17.6 µL	44 µL	88 µL
8260 Calibration Gas Low - 25 mg/L	1.8 µL	8.8µL	17.6 µL	44 µL	88 µL
8260 Calibration Added Low - Mixed	1.8 µL	8.8µL	17.6 µL	44 µL	88 µL
Internal Standard Low - 25 mg/L	8.8 µL	8.8 µL	8.8 µL	8.8 µL	8.8 µL
Final Volume	44 mL	44 mL	44 mL	44 mL	44 mL
FINAL CONCENTRATION in µg/L					
All analytes except as listed below:	1	5	10	25	50
1,4-Dioxane	51	250	500	1250	2500
Isobutyl alcohol	51	250	500	1250	2500
Propionitrile	4	20	40	100	200
Tetrahydrofuran	14	70	140	350	700
Acrolein	5	25	50	125	250
Acetone	5	25	50	125	250
4-Methyl-2-pentanone	5	25	50	125	250
Methyl ethyl ketone	5	25	50	125	250
Tetrahydrofuran	5	25	50	125	250
2-Hexanone	5	25	50	125	250
Internal Standards	5	5	5	5	5

Additionally, for manual injections these standards may be prepared in a 5 mL gas tight syringe and spiked with the appropriate volumes to achieve the analyte concentrations specified above.

7.3.3. Preparation of Initial Calibration Verification (ICV) – Second Source Standard

The ICV must be obtained from a different source than that which is used to prepare the calibration curve, or if one is not available, a second lot of the same manufacturer. The volatile water ICV is prepared in 44 mL vials. All standards are spiked directly through the septum of the 44 mL vial. Prepare the routine level water and medium level soil ICV as follows:

ROUTINE LEVEL ICV	
8260 Calibration Standard - Mixed	22 μL
8260 Calibration Gas - 100 mg/L	22 μL
8260 Calibration Added - 100 mg/L	22 μL
Internal Standard - 50 mg/L	44 μL
Surrogate Standard - 50 mg/L	44 μL
Final Volume	44 mL
FINAL CONCENTRATION in μg/L	
All analytes except as listed below:	50
1,4-Dioxane	2500
Isobutyl alcohol	2500
Propionitrile	200
Tetrahydrofuran	500
Internal Standards	50

Prepare the low-level soil ICV as follows:

LOW LEVEL SOIL ICV	
8260 Calibration Standard - Mixed	2.5 μL
8260 Calibration Gas - 100 mg/L	2.5 μL
8260 Calibration Added - 100 mg/L	2.5 μL
Internal Standard - 50 mg/L	5 μL
Surrogate Standard - 50 mg/L	5 μL
Final Volume	5 mL
FINAL CONCENTRATION in μg/L	
All analytes except as listed below:	50
1,4-Dioxane	2500
Isobutyl alcohol	2500
Propionitrile	200
Tetrahydrofuran	500
Internal Standards	50

Prepare the low level water ICV as follows:

LOW LEVEL WATER ICV	
8260 Calibration Standard Low - Mixed	17.6 µL
8260 Calibration Gas Low - 25 mg/L	17.6 µL
8260 Calibration Added Low - Mixed	17.6 µL
Internal Standard Low - 25 mg/L	8.8 µL
Surrogate Standard - 25 mg/L	8.8 µL
Final Volume	44 mL
FINAL CONCENTRATION in µg/L	
All analytes except as listed below:	10
1,4-Dioxane	500
Isobutyl alcohol	500
Propionitrile	40
Tetrahydrofuran	140
Acrolein	50
Acetone	50
4-Methyl-2-pentanone	50
Methyl ethyl ketone	50
Tetrahydrofuran	50
2-Hexanone	50
Internal Standards	5

Additionally, for manual injections these standards may be prepared in a 5 mL gas tight syringe and spiked with the appropriate volumes to achieve the analyte concentrations specified above.

7.3.4. Preparation of Continuing Calibration Verification (CCV)

CCVs are prepared in the same manner as the calibration standards using the same source that is used to prepare the calibration curve. Prepare the CCVs for routine level water and medium level soil, low-level soil, and low-level water exactly as the Level 2, 3 or 4 standard is prepared in Section 7.3.2.

Additionally, for manual injections these standards may be prepared in a 5 mL gas tight syringe and spiked with the appropriate volumes to achieve the analyte concentrations specified above and injected into the purge vessel.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 Sample collection and preservation for aqueous samples is described in STL SOP LM-MV-5030 and for solid samples in STL SOP LM-MV-5035.

8.2 Unless specified by contract, other regulation, or Quality Assurance Project Plan, the volatile holding times as specified by method and federal regulation are as follows:

Sample Type	Holding Time
Aqueous Preserved (HCl to pH<2)	14 days from collection
Aqueous Non-Preserved	7 days from collection
TCLP Leachate	14 days from leaching
Soil in Sodium Bisulfate solution	14 days from collection
Soil in Encore Device	Extruded into sodium bisulfate within 48 hours, analyzed 14 days from collection
Methanol Extract of Soil	14 days from collection
Sludge	14 days from collection

8.3 Samples are stored at $4^{o}C \pm 2^{o}C$ in a storage area free of organic solvent vapors and direct or intense light. The pH of samples is recorded, and samples may be screened.

8.4 Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 The minimum frequency requirements, acceptance criteria and recommended corrective action for all QC samples are summarized in Section 18, Table 5. Below is a summary of each type of QC sample that is analyzed with the method.

9.2 A Method Blank (MB) and Laboratory Control Sample (LCS) are prepared with each batch of 20 or fewer samples run within a 12-hour window. These samples show that the laboratory is in control, independent of the sample matrix.

9.3 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) are prepared with each batch of 20 samples. Project specific MS/MSD are performed per client request. Sample Duplicates (SD) are performed per client request. These samples show the effect of the sample matrix on the accuracy and precision of the method.

9.4 Surrogate standards are added to all field and QC samples before preparation and/or analysis to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix.

9.5 Internal Standards – All samples are spiked with internal standards as described in Section 7.

9.6 Instrumental QC standards include a Bromofluorobenzene (BFB) every 12 hours, before each Initial Calibration (ICAL) and CCV. A five-point ICAL is generated for each target analyte. After the ICAL, an ICV standard, also referred to as a second source standard, is analyzed to verify the ICAL standard formulation. CCV standards are analyzed every 12 hours immediately following the BFB.

10.0 CALIBRATION AND STANDARDIZATION

10.1 4-Bromofluorbenzene (BFB) - Prior to the acquisition of a calibration curve or the analysis of samples, a 2 μL aliquot of BFB (25 μg/mL) is manually introduced into the GC. The data processing system acquires and averages three scans (apex scan, scan prior, and scan preceding) and performs background subtraction of the single scan prior to the elution of the BFB. The BFB must meet the criteria in Table 2 before initial or continuing calibration may proceed.

10.2 Samples may be run for 12 hours after successful initial and/or continuing calibration. The official start time of the 12-hour window is the time of the BFB injection. The last sample in the window must be injected within 12 hours of that time.

10.3 Initial Calibration – A five-point calibration curve is analyzed at the concentrations and in the manner specified in Section 7.3.2.

10.4 For each target analyte and surrogate, calculate the mean Response Factor (RF) from analyses of the five calibration solutions. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (%RSD - see Appendix B for calculations).

The following criteria must be met for a calibration to be considered acceptable:

- System Performance Check Compounds (SPCCs) must meet the following minimum mean RF: Chloromethane, 1,1-Dichloroethane, and Bromoform - 0.10; Chlorobenzene and 1,1,2,2-Tetrachloroethane – 0.30.
- The following Calibration Check Compounds (CCCs) must have a %RSD of $\leq$ 30%: 1,1-Dichloroethene, Toluene, Chloroform, Ethylbenzene, 1,2-Dichloropropane, and Vinyl chloride. If the CCCs are not included in the list of analytes, then the criteria in Section 10.5 should be used.
- The Relative Retention Time (RRT) for each target analyte in each calibration standard should agree within 0.06 RRT units (See Appendix B for calculation).

10.5 If the %RSD $\leq$ 15%, for all analytes, the calibration is acceptable and the mean RF may be used for quantification. If this criterion is not met, either use another suitable quantification method, or correct the problem and repeat the calibration.

10.6 Alternate Quantification. In some cases, it may be preferable to use either linear regression or Quadratic Equations to quantify the compounds. The following approaches may be used:

Linear Regression - A curve of concentration vs. peak area is generated for each analyte and the correlation coefficient is calculated. The calibration must have a correlation coefficient (r) $\geq$ 0.99 (0.995 for DoD) for acquisition of samples to continue. The use of linear regression requires a minimum of 5 calibration points. See SW-846 Method 8000B for linear regression calculations.

Quadratic Equation - For some compounds, the response is not linear, and in this case a quadratic equation may be employed. For those compounds, the coefficient of determination (r^2) is generated. The coefficient of determination must be $\geq$ 0.99 for acquisition to continue. The uses of quadratic equations require a minimum of 6 calibration points for second order regression and 7 points for third order regression. As chloroethane often follows a quadratic, rather than a linear, pattern, It is anticipated that a sixth point will be analyzed containing only the gases, and that only the chloroethane point is going to be used from this standard. See SW-846 Method 8000B for quadratic equation calculations.

Once a method of calibration is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is generated.

10.7 ICV – Second Source Standard

After each calibration, verify the accuracy of the initial calibration by analyzing the ICV. The calculated concentration of each analyte must be within $\pm$ 25% of the theoretical concentration. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate.

If after successful analysis of the ICV time remains in the 12 -hour analytical window samples may be analyzed without analysis of a CCV; otherwise a CCV must be performed.

10.8 Continuing Calibration Verification (CCV)

At the beginning of each 12-hour shift in which samples are to be run, analyze a BFB as outlined in Section 10.1. A CCV standard, at or below mid-calibration range, is analyzed at the beginning of each 12-hr work shift. The concentration of the CCV is varied.

Calculate the RF and percent difference or drift (see Appendix B for calculation) for each target analyte and surrogate standard. The SPCCs must meet the criteria outlined in Section 10.3. The percent difference or drift for the CCCs must be $\leq$ 20%. If the CCC's 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.

In addition, the internal standard retention time should not change by more than 30 seconds from the retention time in the mid-point standard of the most recent ICAL. The extracted ion current profile (EICP) area of the internal standards in the calibration verification standard should not change by more than a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent ICAL.

If the CCV fails, it may be repeated once. If it still fails, corrective action must be taken. The sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples analyzed after a failing CCV must be reanalyzed, unless the analyte in the CCV is high and that analyte is not

detected in the associated samples.

10.9 Troubleshooting: the following items can be checked in case of calibration, QC or instrument failures:

- Chloromethane response can be low if the purge flow is too fast.
- Bromoform response can be low if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantification 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.
- Contaminated transfer lines in purge-and-trap systems and/or active sites in the trap can degrade the response of Tetrachloroethane and 1,1-dichloroethane.
- 2-Chloroethylvinylether response can be drastically affected/suppressed by soil, foam, or other artifacts contaminating the inside of the soil purge needle. It is also susceptible to active sites/contamination anywhere in the helium path from the autosampler (soils) to the injection port on the GC.
- If the response of the later eluting compounds is low, especially with soils, the purge flow may have been reduced by an obstruction in the helium flow path.
- Poor chromatography and response of the gases are often the result of incorrect placement of the column head in the injection port and/or contamination of the first 6-10 inches of the column from samples and small pieces of injection port septum.
- Erratic response of various compounds and unstable calibrations can be the result of a worn out/contaminated purge trap. Variable matrices such as tissues, soils and moderately foamy samples can be the cause. Samples high in late eluting hydrocarbons or sulfur dioxide will also degrade the trap.

11.0 PROCEDURE

11.1 The following samples are routinely prepared with each batch of samples to be run within a 12 hour window:

11.1.1. Laboratory Method Blanks

For routine level waters, a 44 mL sample vial is filled with reagent water (no air bubbles). Internal and surrogate standards are added separately by the injection through the septum of 44 μL of each of the 50-mg/L internal and surrogate standards.

For medium level soils, a 44 mL sample vial is partially filled with reagent water, 880 μL of methanol is added to it, and the vial is filled with reagent water (no air bubbles). Internal and surrogate standards are added separately by the injection through the septum of 44 μL of each of the 50-mg/L internal and surrogate standards.

For the low-level waters, a 44 mL sample vial is filled with reagent water (no air bubbles). Internal and surrogate standards are added separately by the injection through the septum of 8.8 μL of each of the 25-mg/L internal and surrogate standards.

For low-level soils, a 44 mL sample vial containing 5 gms of sand, 5 mLs of reagent water, and a stir bar is spiked with 5 μL of each of the 50-mg/L internal and surrogate standards.

11.1.2. Laboratory Control Sample (LCS)

For routine level waters and medium level soils, a 44 mL sample vial is filled with reagent water (no air bubbles). Internal and surrogate standards are added separately by the injection through the septum of 44 μL of each of the 50-mg/L internal and surrogate standards, and target analytes are added by injection through the septum of 22 μL of each of the Calibration Standard Mix, Calibration Gas, and Calibration Added standards.

For the low-level waters, internal and surrogate standards are added separately by the injection through the septum of 8.8 μL of each of the 25-mg/L internal and surrogate standards, and target analytes are added by injection through the septum of 17.6 μL of each of the Calibration Standard Mix Low, Calibration Gas Low, and Calibration Added Low standards.

For low-level soils, 5 grams of clean sand is added to a 44 mL sample vial containing a stir bar. The vial is spiked through the septum with 5 μL of each of the 50-mg/L internal and surrogate standards, and 2.5 μL of each of the Calibration Standard Mix, Calibration Gas, and Calibration Added standards.

Additionally, for manual injections the LCS may be prepared in a 5 mL gas tight syringe and spiked with the appropriate volumes to achieve the analyte concentrations specified above.

11.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

MS/MSD are analyzed with each batch of 20 client specific samples of the same matrix. If there is no designated MS/MSD in a batch, one will be prepared if the extra volume for doing so is provided by the client. If not, this is noted as a non-conformance. For DoD, project specific MS/MSD must be run in each batch.

For routine and low-level water samples, and medium level soils, the MS/MSD are prepared exactly as the LCS is prepared in Section 7.5, except that instead of spiking 44 mL of reagent water, a client sample is spiked.

For low-level soil samples, the MS/MSD are prepared by spiking a vial with 5 gms of client sample and 5 mLs of sodium bisulfate solution with 5 μL of each of the 50-mg/L internal and surrogate standards, and 2.5 μL of each of the Calibration Standard Mix, Calibration Gas, and Calibration Added standards through the septum.

Additionally, for manual injections these standards may be prepared in a 5 mL gas tight syringe and spiked with the appropriate volumes to achieve the analyte concentrations specified above.

11.2 Each sample to be analyzed is spiked with internal and surrogate standards. For routine level water and medium level soil samples. Internal and surrogate standards are added separately by the injection through the septum of 44 μL of each of the 50-mg/L internal and surrogate standards. For low level soils, internal and surrogate standards are added separately by the injection through the septum 5 μL of each of the 50-mg/L internal and surrogate standards For low-level waters, internal and surrogate standards are added separately by the injection through the septum of 8.8 μL of each of the 25-mg/L internal and surrogate standards.

11.3 Cleaning blanks (CBLK) consisting of VOA free water may be analyzed after high-level samples at the discretion of the analyst.

11.4 Sample Introduction and Purging - See preparation and introduction Methods 5030 and 5035.

11.5 Gas Chromatography/Mass Spectrometry - Data is acquired and stored over the nominal mass range of 35-300 atomic mass units (amu) with a total cycle time (including scan overhead time) of one second at 70 electron volts. The cycle time is adjusted to measure five or more spectra during the elution of each GC peak. A multi-stage temperature ramp is used to separate the components of interest for this analysis. A typical GC temperature program is described below, but is subject to change at the discretion of the analyst:

Initial temperature:	40° C
Initial time:	4 min.
Ramp1:	7° C/min. to 100° C.
Ramp2:	4.2° C/min. to 120° C, hold for 0 min.
Ramp3:	28° C/min. to 220° C, hold for 2.1 min.
Carrier Gas:	Helium

11.6 Instrument control and acquisition parameters are defined on the ChemStation software for each instrument. Arrange the samples in the autosampler. Acquire the data and evaluate the results to confirm qualitative identification and quantification.

11.7 The data system tentatively identifies target analytes by comparing the retention time of the peaks to a window set around the daily calibration standard, and searches in that area for the primary and up to two secondary ions characteristic of the target analyte. All tentative identifications made by the computer are reviewed and either accepted or rejected by the analyst and/or data reviewer using the following criteria:

- The target analyte is identified by comparison of its background subtracted mass spectrum to a reference spectrum in the user-created database. In general, all ions

that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and their relative abundances should agree within 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance if a tentative identification is to be made, and should be evaluated even if they are below 10% relative abundance.

- The GC retention time for the target analyte should be within 0.06 RRT units of the daily standard.

11.8 Identification requires expert judgment when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components. When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. Because purgeable organic compounds are relatively small molecules and produce comparatively simple mass spectra, this is not a significant problem for most method analytes.

11.9 Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs. Two of the three isomeric xylenes are examples of structural isomers that are not resolved on the capillary column. These groups of isomers will be reported as isomeric pairs.

11.10 Tentatively Identified Compounds (TICs)

TICs may be reported upon client request. In general, TICs whose peak heights are > 10% of the nearest internal standard (>40% for low level water analysis) may be reported.

Perform the library search, and visually compare the sample spectra with the nearest library search and assign a tentative identification. The library search should not include peaks that are < 10% of the nearest internal standard, target analytes, or peaks that elute earlier than 30 seconds before the first target analyte.

The following criteria are used in qualitatively identifying these compounds:

- Relative intensities of ions greater than 10% of the most abundant ion in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%.

- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

The laboratory may determine that a more general identification can be made, such as "unknown alkane, naphthalene derivative, unknown aldehyde, etc.". TIC concentrations are calculated as outlined in Appendix B using an RF of 1.00. All TICs concentrations are reported with a "J" qualifier to indicate that the quantification is estimated. All cases of tentative identification are flagged with an "N" to indicate that there is presumptive evidence of a compound.

11.11 Quantification of Target Analytes

After a compound has been identified, the data system quantifies the concentration of the target compound based on the integrated abundance of the characteristic ion from the EICP using the equations given in Appendix B. If there is matrix interference with the primary ion, a secondary ion may be used for quantification by calculating a mean RF factor for that ion and using that ion to quantify the analyte in the sample. When secondary ion calculations are required, include this information in the non-conformance report and project narrative.

11.12 If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP LP-LB-0006 *Manual Integration.*

11.13 After analysis is complete, evaluate the results against the performance criteria given in Section 10 and Table 3, Section 18 and perform corrective action as necessary.

11.14 Review the samples for carry-over from high-level samples run just prior to the sample for any sign of carry over. Re-analyze the sample is carry-over is suspected.

11.15 Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should ideally result in a determination within the upper half of the calibration curve.

12.0 CALCULATIONS

See Appendix B.

13.0 DATA ASSESSMENT, CORRECTIVE ACTION & REPORTING

13.1 Review the samples, standards and QC samples against the acceptance criteria in Table 4. If the results do not fall within the established limits, perform the recommended corrective action. If corrective action is unsuccessful, document the situation with a nonconformance report and/or qualify the data using an appropriate data qualifier (see

Appendix C for data qualifier definitions). For additional guidance regarding the laboratory's protocol and required elements for each level of data review refer to laboratory SOP LP-LB-003 *Data Review*.

13.2 In the absence of project specific requirements, use the control limits specified in Table 4. The control limits in Table 1 are based on in-house statistically generated limits. In some cases, the in-house limits were outside of Department of Defense (DoD) limits as specified in the Quality Systems Manual for Environmental Laboratories. Where this is the case, the laboratory uses the stricter, DoD limits that are presented in bold in Table 4. For DoD projects, the in-house laboratory limits are also included in the project report.

Based on the number of analytes in the LCS (70-91), it is statistically likely that at least four analytes will marginally exceed the control limits; therefore, 4 marginal exceedances are allowed. A marginal exceedance (ME) is defined as being outside of the control limit of $\pm$ 3 SD, but within $\pm$ 4 SD. In order to easily calculate these limits, the following equation may be used:

$$\text{ME (Lower Limit)} = \text{Lower Limit} - \frac{(\text{Upper Limit} - \text{Lower Limit})}{6}$$

$$\text{ME (Upper Limit)} = \text{Upper Limit} + \frac{(\text{Upper Limit} - \text{Lower Limit})}{6}$$

In addition, the following analytes have been identified as poorly performing analytes based on statistical data accumulated by the laboratory: acrolein in the medium level water and medium level soil matrices, and methyl iodide in the low level water matrix. Decisions regarding batch acceptability are not based on those analytes in the specific matrix identified.

13.3 Data Reporting

The laboratory's RL for each target analyte is provided in Table 1. Report the data to the RL adjusted for sample matrix, percent moisture, and sample dilution/concentration. The reporting limit is the threshold value below which results are reported as non-detected. Report sample results that have concentrations for a target analytes less than the RL with a "U" qualifier. Unless otherwise specified, report the results for solid matrices on a dry weight basis.

Some projects may require reporting positively identified target analytes less than the RL. In this case, the analyte can be qualitatively detected but not accurately quantified. Flag all results less than the RL with a "J" data qualifier.

Some projects may require RLs that are less than the laboratory's routine RL. Sample results may be reported to the project RL if the project RL is greater than the Quantification Limit (QL) and above the MDL. In this context, the QL is defined as the concentration of the low calibration standard. If the project RL is less than the QL, all values less than the QL must be reported as estimated and qualified with a "J".

Further guidance on the application and use of the MDL, RL, and QL is provided in laboratory SOP LP-LB-009 *Determination of Method Detection Limits.*

13.4 Reporting qualifiers are as follows:

B = Analyte is found in the associated method blank as well as the sample
D = Compound is identified in an analysis at a secondary dilution factor
E = Compound quantification is above the instrument's calibration range for this analysis
J = Indicates an estimated quantification value
U = Compound was analyzed for but not detected
X = The reported compound is a suspected laboratory contaminant
Y = an additional qualifier which will be defined at the time of use by the data reviewer
Z = The reported result is based on the combined responses from coeluting compounds
* = Data outside of control limits

13.5 Data Management and Records: All electronic and hardcopy data is managed, retained, and archived as specified in laboratory SOP LP-QA-0014 *Laboratory Records*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009 *Method Detection Limits.*

14.2 A demonstration of analyst capability (IDOC) is required before use of this SOP and any time there is a significant change in instrument type, personnel or test method.

14.3 Employee Training, and IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

14.4 The laboratory statistically derived control limits used to evaluate accuracy, precision and surrogate recoveries are provided in Table 2. The control limits for accuracy are based on compiled data and are set at 3 standard deviations around the mean using the procedures described in laboratory SOP LP-QA-012 *Control Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 The following waste streams are produced when this method is carried out.

- Aqueous Waste
- Solvent Waste
- Solid Waste

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (Method 8260B), Revision 2, December 1996, USEPA SW-846 Methods for Evaluating Solid Waste, Update III.

17.0 SOP REVISION HISTORY

The following changes were made in this revision:

Section 6:	Added computer hardware and software.
Section 7:	7.1 - Removed solvents not used in analytical method.
Section 10:	10.3 - Added additional quantification options. 10.4 - 10.6 Added detail about repeating CCV. 10.7 Added Troubleshooting.
Section 12:	Moved calculations to Appendix B. Moved data reporting to Section 13.
Section 13:	13.1 - Added detail regarding the use of DoD LCS and Surrogate Limits. 13.3 - Added SOP reference for Data Management & Records.
Section 14:	Completely revised section.
Section 17:	New Section added.
Table 1:	Added Footnotes.
Table 3:	Changed Table 3 from Calibration Criteria to Primary Materials Used.
Table 4:	Combined Table 4, 5, and 6, updated control limits, added Low Level Water and Soil Control Limits, added footnotes.
Appendix B:	New Appendix added containing all calculations.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

Table 1:	Target Analyte List, Chemical Abstract Services Numbers and Reporting Limits
Table 2:	BFB Key Ions and Ion Abundance Criteria
Table 3:	Primary Materials Used
Table 4:	Control Limits as Accuracy (%R) and Precision (RPD)
Table 5:	QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action
Appendix A:	Standard Preparation Tables
Appendix B:	Equations
Appendix C:	Terms & Definitions

Table 1: Target Analyte List, Chemical Abstract Services Numbers and Reporting Limits

Analyte	CAS No.	Reporting Limit[1]		
		Water or Soil µg/L or µg/Kg	Low Water µg/L	Methanol Extracts[2] µg/Kg
Acetone	67-64-1	5.0	5.0	500
Acrolein	107-02-8	5.0	5.0	500
Acrylonitrile	107-13-1	5.0	1.0	500
Allyl Chloride	107-05-1	5.0	1.0	500
Benzene	71-43-2	5.0	1.0	500
Bromobenzene	108-86-1	5.0	1.0	500
Bromochloromethane	74-97-5	5.0	1.0	500
Bromodichloromethane	75-27-4	5.0	1.0	500
Bromoform (SPCC)	75-25-2	5.0	1.0	500
Bromomethane	74-83-9	5.0	1.0	500
2-Butanone	78-93-3	5.0	5.0	500
n-Butylbenzene	104-51-8	5.0	1.0	500
sec-Butylbenzene	135-98-8	5.0	1.0	500
tert-Butylbenzene	98-06-6	5.0	1.0	500
Carbon Disulfide	75-15-0	5.0	1.0	500
Carbon Tetrachloride	56-23-5	5.0	1.0	500
Chlorobenzene (SPCC)	108-90-7	5.0	1.0	500
Chloroethane	75-00-3	5.0	1.0	500
2-Chloroethyl Vinyl Ether	110-75-8	5.0	1.0	500
Chloroform (CCC)	67-66-3	5.0	1.0	500
Chloromethane (SPCC)	74-87-3	5.0	1.0	500
Chloroprene	126-99-8	5.0	1.0	500
2-Chlorotoluene	95-49-8	5.0	1.0	500
4-Chlorotoluene	106-43-4	5.0	1.0	500
1,2-Dibromo-3-chloropropane	96-12-8	5.0	1.0	500
Dibromochloromethane	124-48-1	5.0	1.0	500
1,2-Dibromoethane	106-93-4	5.0	1.0	500
Dibromomethane	74-95-3	5.0	1.0	500
1,2-Dichlorobenzene	95-50-1	5.0	1.0	500
1,3-Dichlorobenzene	541-73-1	5.0	1.0	500
1,4-Dichlorobenzene	106-46-7	5.0	1.0	500
cis-1,4-Dichloro-2-butene	1476-11-5	5.0	1.0	500
trans-1,4 Dichloro-2-butene	110-57-6	5.0	1.0	500
Dichlorodifluoromethane	75-71-8	5.0	1.0	500
1,1-Dichloroethane (SPCC)	75-34-3	5.0	1.0	500
1,2-Dichloroethane	107-06-2	5.0	1.0	500
1,1-Dichloroethene (CCC)	75-35-4	5.0	1.0	500
cis-1,2-Dichloroethene	156-59-2	5.0	1.0	500
trans-1,2-Dichloroethene	156-60-5	5.0	1.0	500
1,2-Dichloropropane (CCC)	78-87-5	5.0	1.0	500
1,3-Dichloropropane	142-28-9	5.0	1.0	500
2,2-Dichloropropane	594-20-7	5.0	1.0	500
1,1-Dichloropropene	563-58-6	5.0	1.0	500

Analyte	CAS No.	Reporting Limit[1]		
		Water or Soil µg/L or µg/Kg	Low Water µg/L	Methanol Extracts[2] µg/Kg
cis-1,3-Dichloropropene	10061-01-5	5.0	1.0	500
trans-1,3-Dichloropropene	10061-02-6	5.0	1.0	500
1,4-Dioxane	123-91-1	250	50	25,000
Ethyl Methacrylate	97-63-2	5.0	1.0	500
Ethylbenzene (CCC)	100-41-4	5.0	1.0	500
Freon TF	76-13-1	5.0	1.0	500
Hexachlorobutadiene	87-68-3	5.0	1.0	500
2-Hexanone	591-78-6	5.0	5.0	500
Isobutyl alcohol	78-83-1	250	50	25,000
Isopropylbenzene	98-82-8	5.0	1.0	500
4-Isopropyltoluene	99-87-6	5.0	1.0	500
Methacrylonitrile	126-98-7	5.0	1.0	500
Methyl Iodide	74-88-4	5.0	1.0	500
Methyl Methacrylate	80-62-6	5.0	1.0	500
4-Methyl-2-pentanone	108-10-1	5.0	5.0	500
Methyl-t-Butyl Ether	1634-04-4	5.0	1.0	500
Methylene Chloride	75-09-2	5.0	1.0	500
Naphthalene	91-20-3	5.0	1.0	500
Propionitrile	107-12-0	20	4.0	2,000
n-Propylbenzene	103-65-1	5.0	1.0	500
Styrene	100-42-5	5.0	1.0	500
1,1,1,2-Tetrachloroethane	630-20-6	5.0	1.0	500
1,1,2,2-Tetrachloroethane (SPCC)	79-34-5	5.0	1.0	500
Tetrachloroethene	127-18-4	5.0	1.0	500
Tetrahydrofuran	109-99-9	50	14	5,000
Toluene (CCC)	108-88-3	5.0	1.0	500
1,2,3-Trichlorobenzene	87-61-6	5.0	1.0	500
1,2,4-Trichlorobenzene	120-82-1	5.0	1.0	500
1,2,4-Trimethylbenzene	95-63-6	5.0	1.0	500
1,3,5-Trimethylbenzene	108-67-8	5.0	1.0	500
1,1,1-Trichloroethane	71-55-6	5.0	1.0	500
1,1,2-Trichloroethane	79-00-5	5.0	1.0	500
Trichloroethene	79-01-6	5.0	1.0	500
Trichlorofluoromethane	75-69-4	5.0	1.0	500
1,2,3-Trichloropropane	96-18-4	5.0	1.0	500
Vinyl Acetate	108-05-4	5.0	1.0	500
Vinyl Chloride (CCC)	75-01-4	5.0	1.0	500
Xylene (m,p)	1330-20-7	5.0	2.0	500
Xylene (o)	95-47-6	5.0	1.0	500

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.

[2] Methanol extracts 5 g to 10 mL.

CCC: Calibration Check Compound

SPCC: System Performance Check Compound

Table 2: BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15.0-40.0 percent of mass 95
75	30.0-60.0 percent of mass 95
95	Base peak, 100 percent relative abundance
96	5.0-9.0 percent of mass 95
173	Less than 2.0 percent of mass 174
174	>50.0 percent of mass 95
175	5.0-9.0 percent of mass 174
176	95.0-101.0 percent of mass 174
177	5.0-9.0 percent of mass 176

Table 3: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

[1] Always add acid to water to prevent violent reactions.
[2] Exposure limit refers to the OSHA regulatory exposure limit.

Table 4: Control Limits[1,2] as Accuracy (%R) and Precision[3] (RPD)

Analyte	Low Level Water		Water, Medium Level Soils		Low Level Soils	
	%R	RPD	%R	RPD	%R	RPD
Acetone	80-135	30	60-135	30	45-165	30
Acrolein	65-150	40	20-205*	50*	70-145	35
Acrylonitrile	60-135	35	70-125	30	70-130	30
Allyl Chloride	70-125	30	75-125	30	75-120	30
Benzene	80-125	30	80-120	30	75-120	30
Bromobenzene	80-130	30	80-125	30	75-120	30
Bromochloromethane	80-125	30	80-120	30	75-120	30
Bromodichloromethane	85-130	30	80-135	30	85-130	30
Bromoform	**70-130**	30	**70-130**	30	75-120	30
Bromomethane	45-135	30	40-140	30	50-130	30
2-Butanone	80-145	30	75-135	30	60-160	30
n-Butylbenzene	75-140	30	75-140	30	80-125	30
sec-Butylbenzene	80-125	30	80-130	30	75-120	30
tert-Butylbenzene	80-140	30	75-135	30	75-120	30
Carbon Disulfide	75-120	30	80-125	30	75-120	30
Carbon Tetrachloride	75-120	30	75-120	30	75-120	30
Chlorobenzene	80-120	30	80-125	30	75-120	30
Chloroethane	85-135	30	**60-135**	30	75-120	30
2-Chloroethyl Vinyl Ether	80-125	30	80-125	30	10-220*	50*
Chloroform	80-125	30	75-125	30	75-120	30
Chloromethane	80-130	30	55-130	30	**50-130**	30
Chloroprene	75-120	30	65-140	40	75-120	30
2-Chlorotoluene	80-125	30	80-130	30	75-120	30
4-Chlorotoluene	80-130	30	75-130	30	80-125	30
1,2-Dibromo-3-chloropropane	70-140	30	**60-140**	30	55-125	30
Dibromochloromethane	70-125	30	75-130	30	75-120	30
1,2-Dibromoethane	**80-120**	30	**80-120**	30	75-120	30
Dibromomethane	85-130	30	85-125	30	75-120	30
1,2-Dichlorobenzene	**75-125**	30	**80-130**	30	75-120	30
1,3-Dichlorobenzene	80-130	30	80-130	30	80-125	30
1,4-Dichlorobenzene	85-130	30	**75-125**	30	75-120	30
cis-1,4-Dichloro-2-butene	75-130	30	70-130	30	55-130	30
trans-1,4-Dichloro-2-butene	75-130	30	65-140	35	65-130	30
Dichlorodifluoromethane	80-125	30	60-140	30	50-130	30
1,1-Dichloroethane	75-125	30	80-125	30	75-120	30
1,2-Dichloroethane	80-125	30	**70-130**	30	75-120	30
1,1-Dichloroethene	75-120	30	75-120	30	70-115	30
cis-1,2-Dichloroethene	75-120	30	80-125	30	75-120	30
trans-1,2-Dichloroethene	70-115	30	75-115	30	75-120	30
1,2-Dichloropropane	80-125	30	75-125	30	75-120	30
2,2-Dichloropropane	80-125	30	75-120	30	75-120	30
1,3-Dichloropropane	80-125	30	75-125	30	75-120	30
1,1-Dichloropropene	75-125	30	75-120	30	75-120	30
cis-1,3-Dichloropropene	80-125	30	75-130	30	75-120	30
trans-1,3-Dichloropropene	80-125	30	75-130	30	80-125	30
1,4-Dioxane	80-125	30	65-140	30	70-135	30

Analyte	Low Level Water		Water, Medium Level Soils		Low Level Soils	
Ethyl Methacrylate	80-125	30	75-130	30	75-120	30
Ethylbenzene	80-125	30	80-125	30	75-120	30
Freon TF	75-120	30	75-120	30	75-120	30
Hexachlorobutadiene	85-130	30	70-135	30	80-140	30
2-Hexanone	75-140	30	65-140	30	**45-145**	30
Isobutyl alcohol	70-135	30	55-145	45	70-125	30
Isopropylbenzene	80-130	30	80-130	30	75-120	30
4-Isopropyltoluene	80-125	30	80-130	30	80-125	30
Methacrylonitrile	80-125	30	70-130	30	70-130	30
Methyl Iodide	45-165*	50*	70-145	40	45-140	50
Methyl Methacrylate	75-125	30	75-120	30	70-125	30
4-Methyl-2-pentanone	85-130	30	80-125	30	75-125	30
Methyl-t-Butyl Ether	80-125	30	75-130	30	75-120	30
Methylene Chloride	75-120	30	75-120	30	70-115	30
Naphthalene	**70-155**	30	**55-140**	30	70-135	30
Propionitrile	75-120	30	75-120	30	65-130	30
n-Propylbenzene	80-130	30	80-130	30	75-120	30
Styrene	80-125	30	80-130	30	75-120	30
1,1,1,2-Tetrachloroethane	**80-130**	30	**80-130**	30	75-120	30
1,1,2,2-Tetrachloroethane	**70-135**	30	70-145	30	65-115	30
Tetrachloroethene	65-120	30	50-140	30	75-120	30
Tetrahydrofuran	85-130	30	80-125	30	70-125	30
Toluene	75-120	30	80-125	30	75-120	30
1,2,3-Trichlorobenzene	70-145	30	70-145	30	85-130	30
1,2,4-Trichlorobenzene	**70-140**	30	65-145	30	85-130	30
1,1,1-Trichloroethane	75-120	30	75-120	30	75-120	30
1,1,2-Trichloroethane	**75-130**	30	**75-125**	30	70-115	30
Trichloroethene	75-120	30	75-120	30	75-120	30
Trichlorofluoromethane	80-125	30	75-120	30	70-125	30
1,2,3-Trichloropropane	**75-125**	30	**75-125**	30	65-120	30
1,2,4-Trimethylbenzene	80-125	30	75-125	30	75-120	30
1,3,5-Trimethylbenzene	80-125	30	80-130	30	75-120	30
Vinyl Acetate	70-130	30	60-140	40	45-155	50
Vinyl Chloride	75-130	30	65-135	30	**60-125**	30
Xylene (m,p)	80-125	30	80-125	30	**80-125**	30
Xylene (o)	80-120	30	80-120	30	80-125	30
Surrogate Standards						
4-Bromofluorobenzene	**85-115**	NA	**85-115**	NA	**85-120**	NA
1,2-Dichlorobenzene-d4	80-125	NA	85-125	NA	80-125	NA
1,2-Dichloroethane-d4	80-125	NA	**70-120**	NA	80-125	NA
Toluene-d8	85-125	NA	85-120	NA	85-115	NA

[1] The in-house statistical control limits posted in this table are those in effect on the revision date of this SOP. These limits are subject to change based on performance trends.

[2] Those limits appearing in bold are limits for which the in-house limit it outside of the DoD required limit, and corrective action is taken based on the DoD limit per Section 13.2. However, no DoD limits were specifically calculated for Low level waters.

[3] RPD for MS/MSD only.

[*] Identified as poorly performing analyte. Decisions regarding batch acceptability are not based on analyte in the specific matrix indicated.

Table 5: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Minimum Frequency	Acceptance Criteria	Recommended Corrective Action[1]
BFB	Before initial and continuing calibration, every 12 hours	See Table 2	Reshoot, retune mass spectrometer
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CCCs: %RSD $\leq$ 30% SPCCs: mean RF per Section 10.4. Linear Regression: r $\geq$ 0.99 (0.995 for DoD) Quadratic: $r^2 \geq 0.99$	Correct problem and repeat initial calibration.
ICV	After each initial calibration	%Difference ± 25%	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Beginning of each 12-hour window, as established by a compliant BFB.	SPCCs: must meet minimum RF CCCs: %D $\leq$ 20%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs at different concentrations pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and samples are non-detects.
MB	One per batch of 20 or fewer samples	< RL DoD: $\leq$ ½ RL for all analytes except < RL for acetone, 2-butanone, and methylene chloride for any sample $\geq$ RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB and samples (if samples have been run), and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, reanalysis may not be required.
LCS	One per batch of 20 or fewer samples	Evaluated against control limits in Table 4, 4 Marginal Exceedances allowed.	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS and samples (if samples have been run), and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch, DoD: project specific per extraction batch SD: Per client request	Evaluated against control limits in Table 4	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze. Flag all reported values outside of control limits.
Surrogate Standard	All field and QC samples	Evaluated against control limits in Table 4	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.
Internal Standard	All field and QC samples	Area between 50-200% of area of daily calibration internal standard area	Same as above.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that data quality is known and documented. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

Appendix A: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory SOP LP-LB-002 *Standard Preparation* for further guidance on the preparation of standard solutions.

All standards are prepared using volumetric glassware including Hamilton syringes. All standards are made with purge and trap grade methanol, demonstrated to be analyte free and standards are stored at -10 to -20°C in amber glass mini-inert vials, except for the routine level water and medium level surrogate and internal standard solutions, which may be stored in volumetric flasks at 2-6°C.

Table Legend:
C_P = Concentration of Parent Standard
V_P = Volume of Parent Standard
V_S = Volume of Prepared Standard
C_S = Theoretical Concentration of Prepared Standard

TUNING STANDARD

Bromofluorobenzene – 25 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromofluorobenzene	30003	5000 µg/mL	125 µL	25 mL	25 mg/L

Expiration Date: 6 months from preparation or expiration of parent, whichever is earlier.

CALIBRATION STANDARDS

Internal Standard Spiking Solution – 50 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Chlorobenzene-d_5 1,4-Dichlorobenzene-d_4 Fluorobenzene	50684	1000 µg/mL	1250 µL	25 mL	50 mg/L

Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

Surrogate Standard – 50 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromofluorobenzene 1,2-Dichlorobenzene-d_4 1,2-Dichloroethane-d_4 Toluene-d_8	53837	2000 µg/mL	625 µL	25 mL	50 mg/L

Restek Catalog #
Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

8260 Calibration – Mixed Concentration

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromofluorobenzene 1,2-Dichlorobenzene-d_4 1,2-Dichloroethane-d_4 Toluene-d_8	Surrogate: 53837	2000 μg/mL	200 μL	4 mL	100 mg/L
Vinyl Acetate	30216	2000 μg/mL	200 μL	4 mL	100 mg/L
Methyl acrylate Methyl methacrylate Allyl chloride Nitrobenzene Acrylonitrile Pentachloroethane Ethyl methacrylate	Mix 7B: 30202B	2000 μg/mL	200 μL	4 mL	100 mg/L
54 volatile Components, see Catalog	Mega-mix: 30431	2000 μg/mL	200 μL	4 mL	100 mg/L
1,4-Dioxane Isobutyl alcohol Tetrahydrofuran Propionitrile trans-1,4-Dichloro-2-butene 1,1,2-Trichlorotrifluoroethane 2-Chloro-1,3-butadiene Carbon disulfide Methacrylonitrile Methyl-tert-butyl ether Iodomethane cis-1,4-Dichloro-2-butene	Custom: 56531	100,000 μg/mL 100,000 μg/mL 18,000 μg/mL 8000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL	200 μL	4 mL	5000 mg/L 5000 mg/L 900 mg/L 400 mg/L 100 mg/L 100 mg/L 100 mg/L 100 mg/L 100 mg/L 100 mg/L 100 mg/L 100 mg/L 100 mg/L

Expiration Date: 2 months from preparation or expiration of parent, whichever is earlier.

8260 Calibration Gas – 100 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromomethane Chloroethane Chloromethane Dichlorodifluoromethane Trichlorofluoromethane Vinyl Chloride	30042	2000 μg/mL	75 μL	1500 μL	100 mg/L

Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

8260 Calibration Added – 100 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
2-Chloroethylvinylether	30265	2000 μg/mL	200 μL	4 mL	100 mg/L
Acrolein	53547	5000 μg/mL	80 μL	4 mL	100 mg/L
Acetone 4-Methyl-2-pentanone Methyl ethyl ketone Tetrahydrofuran 2-Hexanone	Mix 7A: 30202A	2000 μg/mL	200 μL	4 mL	100 mg/L

Expiration Date: 2 months from preparation or expiration of parent, whichever is earlier.

LOW LEVEL WATER CALIBRATION

Internal Standard Low – 25 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Chlorobenzene-d_5 1,4-Dichlorobenzene-d_4 Fluorobenzene	50684	1000 μg/mL	150 μL	6 mL	25 mg/L

Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

Surrogate Standard Low – 25 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromofluorobenzene 1,2-Dichlorobenzene-d_4 1,2-Dichloroethane-d_4 Toluene-d_8	53837	2000 μg/mL	75 μL	6 mL	25 mg/L

Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

8260 Calibration Low – Mixed Concentration

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromofluorobenzene 1,2-Dichlorobenzene-d_4 1,2-Dichloroethane-d_4 Toluene-d_8	Surrogate: 53837	2000 μg/mL	55 μL	4.4 mL	25 mg/L
Vinyl Acetate	30216	2000 μg/mL	55 μL	4.4 mL	25 mg/L
Methyl acrylate Methyl methacrylate Allyl chloride Nitrobenzene Acrylonitrile Pentachloroethane Ethyl methacrylate	Mix 7B: 30202B	2000 μg/mL	55 μL	4.4 mL	25 mg/L
54 volatile Components, see Catalog	Mega Mix: 30431	2000 μg/mL	55 μL	4.4 mL	25 mg/L
1,4-Dioxane Isobutyl alcohol Tetrahydrofuran Propionitrile trans-1,4-Dichloro-2-butene 1,1,2-Trichlorotrifluoroethane 2-Chloro-1,3-butadiene Carbon disulfide Methacrylonitrile Methyl-tert-butyl ether Iodomethane cis-1,4-Dichloro-2-butene	Custom: 56531	100,000 μg/mL 100,000 μg/mL 18,000 μg/mL 8000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL 2000 μg/mL	55 μL	4.4 mL	1250 mg/L 1250 mg/L 225 mg/L 100 mg/L 25 mg/L 25 mg/L 25 mg/L 25 mg/L 25 mg/L 25 mg/L 25 mg/L 25 mg/L 25 mg/L

Expiration Date: 2 months from preparation or expiration of parent, whichever is earlier.

8260 Calibration Gas Low – 25 mg/L

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
Bromomethane Chloroethane Chloromethane Dichlorodifluoromethane Trichlorofluoromethane Vinyl Chloride	30042	2000 μg/mL	19 μL	1520 μL	25 mg/L

Expiration Date: 1 month from preparation or expiration of parent, whichever is earlier.

8260 Calibration Added Low – Mixed Concentration

Analyte	Restek Catalog #	C_P	V_P	V_S	C_S
2-Chloroethylvinylether	30265	2000 μg/mL	55 μL	4.4 mL	25 mg/L
Acrolein	53547	5000 μg/mL	110 μL	4.4 mL	125 mg/L
Acetone 4-Methyl-2-pentanone Methyl ethyl ketone Tetrahydrofuran 2-Hexanone	30202A	2000 μg/mL	275 μL	4.4 mL	125 mg/L

Expiration Date: 2 months from preparation or expiration of parent, whichever is earlier.

Appendix B: Equations

Response Factor (RFx) = $\frac{\text{Area}_x \text{ X Concentration}_{is}}{\text{Area}_{is} \text{ X Concentration}_x}$

Where: x=compound, is = Internal Standard

Relative Retention Time (RRT) = $\frac{\text{Retention Time}_x}{\text{Retention Time}_{is}}$

Where: x=compound, is = Internal Standard

Mean Response Factor ($\overline{RF}$) = $\frac{\sum_{i=1}^{n} RF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Response Factor (SD) = $\sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Response = $\frac{SD}{\overline{RF}} \times 100\%$

Percent Difference (%D) = $\frac{RF_v - \overline{RF}}{\overline{RF}} \times 100\%$

where: RF_v = Response Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $\frac{\text{Calculated Concentration} - \text{Theoretical Concentration}}{\text{Theoretical Concentration}}$ X 100%

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD) = $\frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Water

$$C_x = \frac{A_x \times C_{is}}{A_{is} \times \text{Mean RF}} \times DF$$

Solids

$$C_x = \frac{A_x \times C_{is}}{A_{is} \times \text{Mean RF} \times \text{Percent Solids}} \times DF$$

Where

C_x	=	Concentration of compound (µg/L)
A_{is}	=	Area of quantification ion for associated internal standard.
A_x	=	Area of quantification ion for compound.
C_{is}	=	Concentration of associated internal standard (µg/L).
DF	=	Dilution Factor.
Mean RF	=	Mean Response Factor from initial calibration, or 1 for a tentatively identified compound

Appendix C: Terms & Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed.

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Calibration Check Compounds (CCCs): Selective analytes from the compound list that are used 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.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Internal Standard: a known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery for each analyte.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

System Performance Check Compounds (SPCCs): Selective analytes from the compound list that are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system.

STANDARD OPERATING PROCEDURE
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS
SW-846 Method 8270C
Matrix: Non-Potable Water, Solid & Chemical Materials, Tissue

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APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the laboratory procedure used to determine the concentration of semivolatile organic compounds in extracts derived from non-potable water, solid and chemical materials, and tissue. This SOP is applicable to the analytical procedure only; the extraction and extract cleanup methods referenced in this SOP are described in the following laboratory SOPs:

LM-OP-3550 Ultrasonic Extraction
LM-OP-3540 Soxhlet Extraction
LM-OP-3510 Separatory Funnel Extraction
LM-OP-GPC Gel Permeation Chromatography (GPC)
LP-OP-3541 Automated Soxtherm Extraction
LM-OP-3580A Waste Dilution

1.2 The analytes that can be determined by this procedure and their associated Reporting Limits (RL) are listed in Table 1A & 1B, Section 18.

1.3 The following problems have been associated with compounds analyzed by this method: dichlorobenzidine and 4-chloroaniline may be subject to oxidative losses during solvent concentration; hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reactions in acetone solution, and photochemical decomposition; and n-nitrosodiphenylamine decomposes in the gas chromatograph inlet forming diphenylamine and, consequently, is detected as diphenylamine. Likewise, 1,2-Diphenylhydrazine is detected as Azobenzene due to decomposition during the GC/MS analysis.

2.0 SUMMARY OF METHOD

2.1 Samples are prepared for analysis by GC/MS using an appropriate extraction method and if necessary, extract cleanup procedure. An aliquot of extract is injected into the gas chromatograph (GC), where it is volatilized in the injection port and swept onto the chromatographic column in which a temperature program is used to separate the target compounds, and they are then detected by a mass spectrometer (MS). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.2 This procedure is based on SW-846 Method 8270C, Revision 3, December 1996.

3.0 DEFINITIONS

A list of terms and definitions is given in Appendix B.

4.0 INTERFERENCES

4.1 Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the extracted ion current profiles (EICPs). All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

4.2 Injection syringes should be adequately flushed with solvent between injections in order to remove all traces of the prior sample.

4.3 Co-extracted Interferences may include lipids, polymers, copolymers, proteins, natural resins, cellular components, viruses, steroids, and high-molecular weight compounds. GPC, which is size exclusion chromatography, is appropriate for cleanup of these types of polar and non-polar interferences.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 Primary Materials Used

Table 2, Section 18 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

- Methylene chloride

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance: Capable of weighing to 0.1 mg.

6.2 Containers

- 2 mL Autosampler vials with 200 uL inserts, PFTE crimp top.
- 4 mL sample vials with PFTE lined screw top caps.

6.3 Computer Hardware/Software: GCMS Acquisition Platform - Hewlett-Packard ChemStations. Data Processing - Hewlett-Packard 9000-series computers, an HP9000 D250 (Chemsvr4) and an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5.

6.4 Instrumentation

- SVOA Autosampler: HP 7673A™, CTC A200S™ or equivalent.
- Gas Chromatograph: Hewlett-Packard™ 5890 GC, 6890 GC or equivalent.
- Mass Spectrometer: Hewlett-Packard™ 5971, 5972and 5973 MSD or equivalent.
- Primary Column: Restek™ RTX-5 30m x 0.25mm ID x .25 um film thickness or equivalent.
- Guard Column: Restek™ Deactivated 5m x 0.25 mm ID or equivalent.
- Column unions: Restek Press-Tights™ or equivalent.
- Injection port liners: Single Goose Neck, borosilicate glass. Restek™ 20799 or equivalent.
- Injection port septa: HP™, 11 mm Thermo Red or equivalent.
- Data System: Hewlett-Packard Chem server™, Target 3.5 processing software and Hewlett-Packard ChemStation software for instrument control and acquisition.

6.5 Syringes: 10 uL, 25 uL, 50 uL, 100 uL, 1000 uL.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Methylene Chloride (CH_2Cl_2), Pesticide quality.

7.2 Standards

Stock standard solutions are purchased from commercial vendors and stored according to manufacturer instructions. The stock standard solutions remain unopened until time of use and are considered acceptable until the expiration date given by the manufacturer. Intermediate and working standards are prepared in the laboratory by dissolving a volume of stock standard solution in an appropriate solvent and diluting to a specified volume. Standard solutions are stored in amber glass vials with Teflon lined screw caps at a temperature of 4^oC ($\pm$ 2). Unless otherwise noted, prepared standard

solutions are assigned an expiration date of 6 months from date of preparation unless the expiration date of the parent standard expires sooner in which case, the expiration date of the parent standard is used.

Calibration Mix (CAL MIX): Dilute appropriate volumes of commercially purchased stock standard solutions in methylene chloride to achieve a final concentration of 166.67 ng/uL for each analyte and surrogate compound.

Working Calibration Standards: Dilute an appropriate volume(s) of the CAL MIX in methylene chloride to achieve a series of standards at the following concentrations: 10, 25, 40, 60, and 80 ng/uL (20, 50, 80, 120, and 160 ng per 2-ul injection)

Internal Standard Solution: Dilute an appropriate volume of the commercially prepared stock standard solution (i.e. Restek™ Internal Standard Mix) in methylene chloride to achieve a concentration of 500 ng/uL.

Initial Calibration Verification (ICV) (25ng/uL): Dilute appropriate volumes of second source stock standard solutions in methylene chloride to achieve a final concentration of 25 ng/uL.

Decafluorotriphenylphosphine (DFTPP) Mix (25ng/uL): Dilute appropriate volumes of commercially prepared stock standard solutions (DFTPP, Benzidine, Pentachlorophenol, and DDT) in methylene chloride to achieve a final concentration of 25 ng/uL.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Sample extracts must be stored at 4°C ± 2° until the time of analysis. The analytical holding time is 40 days from date of sample extraction.

8.2 Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 The minimum frequency requirements, acceptance criteria and recommended corrective action for all QC samples are summarized in Section 18, Table 4. Below is a summary of each type of QC sample that is analyzed with the method.

9.2 A Method Blank (MB) and Laboratory Control Sample (LCS) are prepared with each batch of 20 or fewer samples. These samples show that the laboratory is in control independent of the sample matrix.

9.3 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) are prepared with each batch of 20 samples. Project specific MS/MSD are performed per client request. Sample Duplicates (SD) are performed per client request. These samples show the effect of the sample matrix on the accuracy and precision of the method.

9.4 Surrogate standards are added to all field and QC samples before preparation and/or analysis to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix.

9.5 Internal Standards – All samples are spiked with internal standards as described in Section 7.

9.6 Instrumental QC standards include a DFTPP every 12 hours, before each Initial Calibration (ICAL) and CCV. A five-point ICAL is generated for each target analyte. After the ICAL, an ICV, also referred to as a second source standard, is analyzed to verify the ICAL standard formulation. Continuing Calibration Verification (CCV) standards are analyzed every 12 hours immediately following the DFTPP.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Prior to the acquisition of a calibration curve, CCV, or the analysis of samples, a 2 μL aliquot of DFTPP (25 μg/mL) is injected into the GC. The data processing system acquires and averages three scans (apex scan, scan prior, and scan preceding) and performs background subtraction of the single scan prior to the elution of the DFTPP. The DFTPP must meet the criteria in Table 4 before initial or continuing calibration may proceed. If criteria are not met, retune the instrument. Analysis may not proceed until tune criteria are met.

The DFTPP is also used to evaluate column performance and injection port inertness. The Tailing factor for Benzidine and pentachlorophenol are evaluated and must be less than 5.0 and 3.0, respectively. For DoD projects, DDT breakdown is evaluated. The degradation of DDT must not exceed 20%. If these criteria are not met, perform corrective action before further analysis. Corrective action may include cleaning of the injection port or clipping the column.

10.2 Samples may be run for 12 hours after successful initial and/or continuing calibration. The official start time of the 12-hour window is the time of the DFTPP injection. The last sample in the window must be injected within 12 hours of that time.

10.3 Initial Calibration (ICAL)

Add 4-uL of internal standard solution to each calibration standard to achieve a final internal standard concentration of 20-ng/uL in extract (40-ng on column, 2-uL injection). Inject 2-uL of each calibration standard onto the GC/MS system. Ensure the analysis of the calibration standards is within the 12-hour analytical window that was started at the time of injection of the DFTPP standard.

Calibrate the instrument with a minimum of five calibration standards that include all compounds of interest at each concentration level. The recommended concentration for each calibration level is 10, 25, 40, 60, and 80 ng/uL (20, 50, 80, 120, and 160 ng per 2-ul injection). The 10-ng/uL standard is not used for the following analytes: Benzoic acid, 2,4,5-Trichlorophenol, 2-Nitroaniline, 3-Nitroaniline, 2,4-Dinitrophenol, 4-Nitrophenol, 4-

Nitroaniline, 4,6-Dinitro-2-methylphenol, Pentachlorophenol, and Benzidine. To provide a five-point calibration for these compounds, a 180-ng/uL standard is analyzed. While all analytes are present in the 180-ng/uL, it only needs to be processed for those above named analytes.

If samples are to be analyzed for the extended 8270 Appendix IX target compound list (See Table 1B), use a second set of calibration standards to minimize constituent deterioration due to compound interaction. Analyze two sets each of five calibrations standards (ten total) at the concentrations listed above. Each calibration set includes specific compounds that are determined to be free from interactions of interference.

10.4 For each target analyte and surrogate, calculate the mean Response Factor (RF) from analyses of the five calibration solutions. Calculate the standard deviation (SD) and Percent Relative Standard Deviation (%RSD - see Appendix A for calculations).

The following criteria must be met for a calibration to be considered acceptable:

- System Performance Check Compounds (SPCCs) must meet the following minimum mean RF: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol – 0.050.
- Calibration Check Compounds (CCCs) must have a %RSD of $\leq$ 30%: Acenaphthene, 4-Chloro-3-methylphenol, 1,4-Dichlorobenzene, 2,4-Dichlorophenol, Hexachlorobutadiene, 2-Nitrophenol, N-nitrosodiphenylamine, Phenol, Di-n-octyl phthalate Pentachlorophenol, Fluoranthene, 2,4,6-Trichlorophenol, Benzo(a)pyrene. If the CCCs are not included in the list of analytes, then the criteria in 10.5 should be used.
- The Relative Retention Time (RRT) for each target analyte in each calibration standard should agree within 0.06 RRT units (See Appendix A for calculation).

10.5 If the %RSD $\leq$ 15%, for all analytes, the calibration is acceptable and the mean RF may be used for quantification. If this criterion is not met, either use another suitable quantification method, or correct the problem and repeat the calibration.

10.6 Alternate Quantification. In some cases, it may be preferable to use either linear regression or Quadratic Equations to quantify the compounds. The following approaches may be used:

Linear Regression - A curve of concentration vs. peak area is generated for each analyte and the correlation coefficient is calculated. The calibration must have a correlation coefficient (r) $\geq$ 0.99 (0.995 for DoD) for acquisition of samples to continue. The use of linear regression requires a minimum of 5 calibration points. See SW-846 Method 8000B for linear regression calculations.

Quadratic Equation - For some compounds, the response is not linear, and in this case a quadratic equation may be employed. For those compounds, the coefficient of determination (r^2) is generated. The coefficient of determination must be $\geq$ 0.99 for acquisition to continue. The uses of quadratic equations require a minimum of 6

calibration points for second order regression and 7 points for third order regression. See SW-846 Method 8000B for quadratic equation calculations.

Once a method of calibration is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is generated.

10.7 ICV – Second Source Standard

After each calibration, verify the accuracy of the initial calibration by analyzing the ICV. To prepare the ICV, add 4-uL of internal standard solution to 100-uL of the 25-ng/uL ICV solution, and inject 2-uL of the solution to achieve an on-column concentration of 50-ng. The calculated concentration of each analyte must be within $\pm$ 25% of the theoretical concentration. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate.

If after successful analysis of the ICV time remains in the 12 -hour analytical window, samples may be analyzed without analysis of a continuing calibration verification check standard (CCV); otherwise a CCV must be performed.

10.8 Continuing Calibration Verification (CCV)

At the beginning of each 12-hour shift in which samples are to be run, analyze a DFTPP as outlined in Section 10.1. A CCV standard, at or below mid-calibration range, is analyzed at the beginning of each 12-hr work shift. The concentration of the CCV is varied.

Inject 2 uL of the prepared CCV standard, acquire the data and evaluate the results. If samples are to be analyzed for the extended Appendix IX target compound list (See Table 1B), analyze 2 CCVs containing the same target compounds as established in the initial calibration.

Calculate the RF and percent difference or drift (see Appendix A for calculation) for each target analyte and surrogate standard. The SPCCs must meet the criteria outlined in Section 10.4. The percent difference or drift for the CCCs must be $\leq$ 20%. 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.

In addition, the internal standard retention time should not change by more than 30 seconds from the retention time in the mid-point standard of the most recent ICAL. The EICP area of the internal standards in the calibration verification standard should not change by more than a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent ICAL.

If the CCV fails, it may be repeated once. If it still fails, corrective action must be taken. The analysis may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If the two CCVs do not meet the criteria, recalibration is required prior to running samples. Samples analyzed after a failing CCV must be reanalyzed, unless the analyte in the CCV is high and that analyte is not

detected in the associated samples.

10.9 Troubleshooting: the following items can be checked in case of calibration, QC or instrument failures:

- Benzidine is subject to oxidative loss during extraction and chromatographs poorly, injection port and/or column maintenance may be required.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet, injection port and/or column maintenance may be required.
- N-nitrosodiphenylamine decomposes in the inlet and cannot be separated from diphenylamine. Compound is reported as N-nitrosodiphenylamine.
- 1,2-Diphenylhydrazine decomposes to Azobenzene in the analytical portion of the procedure and as such is reported as Azobenzene.
- Acid compounds are subject erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material, injection port and/or column maintenance may be required.
- Loss of sensitivity for higher boiling compounds and internal standards may be indicative of a leak at the inlet. Replace septa and/or re-tighten lower inlet connection.
- Carryover contamination may indicate empty rinse vials.

11.0 PROCEDURE

11.1 Extract Preparation & Analysis

Allow the extracts to warm to room temperature. Transfer 100-uL of extract to a 1-mL auto-sampler vial with insert. Add 4-uL of the internal standard solution to each vial and seal the vial with a PTFE lined crimp top cap. If an alternate extract volume is used (e.g. 50 uL extract) adjust the volume of internal standard proportionately.

When extract screening has been performed, prepare sample dilutions by diluting an appropriate volume of sample extract in methylene chloride. Serial dilutions may be required if the relative volumes needed for a single dilution step exceed the accuracy of the pipettes. For example, a sample requires a 0.1% analysis in order to have target constituents within the upper half of the calibrated range. This level of dilution in a 100-uL aliquot requires 0.1-uL of sample extract. However, the gradations of the microliter pipette are to 0.2-uL. In this situation, it is necessary to perform a serial dilution of 1:100 (1.0%) and a 10:100 (10%) to achieve an analysis concentration of 0.1% of the original extract.

11.2 Instrument Operating Conditions

Gas Chromatography/Mass Spectrometry - Data is acquired and stored over the nominal mass range of 35-500 atomic mass units (amu) with a total cycle time (including scan overhead time) of one second per scan at 70 electron volts. The cycle time is

adjusted to measure five or more spectra during the elution of each GC peak. A typical GC temperature program is described below, but is subject to change at the discretion of the analyst:

Initial Temperature:	35°C for 2 minutes.
Temperature Program:	35°C to 320°C at 14°/min.
Final Temperature:	320°C for 5.6 min or until Benzo (g,h,i) perylene has eluted
Injector Temperature:	250°C
Transfer Line Temperature:	300°C
Injector:	Grob-like, splitless
Injection volume:	2 μL
Carrier Gas:	Helium

11.3 Instrument control and acquisition parameters are defined on the ChemStation software for each instrument. Arrange the samples in the auto-sampler. Inject 2-uL of samples using the same instrument operating conditions that were used for initial calibration. Acquire the data and evaluate the results to confirm qualitative identification and quantification.

11.4 The data system tentatively identifies target analytes by comparing the retention time of the peaks to a window set around the daily calibration standard, and searches in that area for the primary and up to two secondary ions characteristic of the target analyte. All tentative identifications made by the computer are reviewed and either accepted or rejected by the analyst and/or data reviewer using the following criteria:

- The target analyte is identified by comparison of its background subtracted mass spectrum to a reference spectrum in the user-created database. In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and their relative abundances should agree within 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance if a tentative identification is to be made, and should be evaluated even if they are below 10% relative abundance.
- The GC retention time for the target analyte should be within 0.06 RRT units of the daily standard.

11.5 Identification is hindered when components are not chromatographically resolved from interfering analyte peaks or non-target constituents (background). When chromatographic peaks obviously indicate contribution from more than one component (broadened peak with shoulder(s) or a valley between two or more maxima), examine the EICPs to select the appropriate analyte spectra over the entire peak and use selective background subtraction in order to positively identify target analytes and account for extraneous ions. For coeluting compounds, the identification criteria will be met, but the analyte spectrum will contain extraneous ions contributed by the coeluting compound.

11.6 Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs.

Complex environmental matrices, baseline upsets, coelution and peak shape variation can complicate automatic data system integration causing inaccurate and/or missed identification that should be corrected with manual integration. To assure accurate qualitative identification, optimize the data system integration parameters to ensure consistency in integration between standards and sample and evaluate each chromatogram to verify the identification for each target analyte. If necessary, perform manual integration to correct for data processing or integration error.

11.2.2 Tentatively Identified Compounds (TICs)

TICs may be reported upon client request. In general, TICs whose peak areas are > 10% of the nearest internal standard may be reported.

Perform the library search, and visually compare the sample spectra with the nearest library search and assign a tentative identification. The library search should not include peaks that are < 10% of the nearest internal standard, target analytes, or peaks that elute earlier than 30 seconds before the first target analyte.

The following criteria are used in qualitatively identifying these compounds:

- Relative intensities of ions greater than 10% of the most abundant ion in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

The laboratory may determine that a more general identification can be made, such as “unknown aliphatic compound, unknown aromatic compound, unknown cycloalkane, etc.”. TIC concentrations are calculated as outlined in Appendix A using an RF of 1.00. All TICs concentrations are reported with a “J” qualifier to indicate that the quantification is estimated. All cases of tentative identification are flagged with an “N” to indicate that there is presumptive evidence of a compound.

11.7 Quantification of Target Analytes

After a compound has been identified, the data system quantifies the concentration of the target compound based on the integrated abundance of the characteristic ion from the EICP using the equations given in Appendix A. If there is matrix interference with

the primary ion, a secondary ion may be used for quantification by calculating a mean RF factor for that ion and using that ion to quantify the analyte in the sample. When secondary ion calculations are required, include this information in the non-conformance report and project narrative.

11.8 If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP LP-LB-0006 *Manual Integration.*

11.9 After analysis is complete, evaluate the results against the performance criteria given in Section 10 and Table 3, Section 18 and perform corrective action as necessary.

11.10 Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should ideally result in a determination within the upper half of the calibration curve.

12.0 CALCULATIONS

See Appendix A.

13.0 DATA REVIEW, CORRECTIVE ACTION & REPORTING

13.1 Review the samples, standards and QC samples against the acceptance criteria in Table 3. If the results do not fall within the established limits, perform the recommended corrective action. If corrective action is unsuccessful, document the situation with a nonconformance report and/or qualify the data using an appropriate data qualifier (see Section 13 for data qualifier definitions). For additional guidance regarding the laboratory's protocol and required elements for each level of data review refer to laboratory SOP LP-LB-003 *Data Review*.

13.2 In the absence of project specific requirements, use the in-house statistically derived control limits specified in Table 5A & B. Table 5A also contains specific control limits that are required to be used for DoD projects.

Based on the number of analytes in the LCS (70-91), it is statistically likely that at least four analytes will marginally exceed the control limits; therefore, 4 marginal exceedances are allowed. A marginal exceedence (ME) is defined as being outside of the control limit of $\pm$ 3 SD, but within $\pm$ 4 SD. In order to easily calculate these limits, the following equation may be used:

$$\text{ME (Lower Limit)} = \text{Lower Limit} - \frac{(\text{Upper Limit} - \text{Lower Limit})}{6}$$

$$\text{ME (Upper Limit)} = \text{Upper Limit} + \frac{(\text{Upper Limit} - \text{Lower Limit})}{6}$$

13.3 The following analytes have been identified as poorly performing analytes based on statistical data by both the DoD and the laboratory. Decisions regarding batch

acceptability are not based on those analytes in the specific matrix identified. These are footnoted in Table 4.

Analyte	Based on:
Aqueous Matrix	
4-nitrophenol	DoD and Laboratory Data
Benzoic acid	DoD and Laboratory Data
Phenol	DoD and Laboratory Data
Phenol-d5	DoD and Laboratory Data
Benzidine	Laboratory Data[1]
2-Chlorobenzoic acid	Laboratory Data[1]
Hexachlorocyclopentadiene	Laboratory Data[1]
Pyridine	Laboratory Data[1]
Solids Matrix	
3,3'-Dichlorobenzidine	DoD and Laboratory Data
4-Chloroaniline	DoD and Laboratory Data
Benzoic acid	DoD and Laboratory Data
Aniline	Laboratory Data[1]
Benzidine	Laboratory Data[1]
2-Chlorobenzaldehyde	Laboratory Data[1]
2-Chlorobenzoic acid	Laboratory Data[1]
3-Chlorobenzaldehyde	Laboratory Data[1]
4-Chlorobenzaldehyde	Laboratory Data[1]
Pyridine	Laboratory Data[1]

[1] DoD data not available

13.4 Data Reporting

The laboratory's RL for each target analyte is provided in Table 1A & 1B. Report the data to the RL adjusted for sample matrix, percent moisture, and sample dilution/concentration. The reporting limit is the threshold value below which results are reported as non-detected. Report sample results that have concentrations for a target analytes less than the RL with a "U" qualifier. Unless otherwise specified, report the results for solid matrices on a dry weight basis.

Some projects may require reporting positively identified target analytes less than the RL. In this case, the analyte can be qualitatively detected but not accurately quantified. Flag all results less than the RL with a "J" data qualifier.

Some projects may require RLs that are less than the laboratory's routine RL. Sample results may be reported to the project RL if the project RL is greater than the Quantification Limit (QL) and above the MDL. In this context, the QL is defined as the concentration of the low calibration standard. If the project RL is less than the QL, all values less than the QL must be reported as estimated and qualified with a "J".

Further guidance on the application and use of the MDL, RL, and QL is provided in laboratory SOP LP-LB-009 *Determination of Method Detection Limits.*

13.5 Reporting qualifiers are as follows:

B = Analyte is found in the associated method blank as well as the sample
D = Compound is identified in an analysis at a secondary dilution factor
E = Compound quantification is above the instrument's calibration range for this analysis
J = Indicates an estimated quantification value
U = Compound was analyzed for but not detected
X = The reported compound is a suspected laboratory contaminant
Y = an additional qualifier which will be defined at the time of use by the data reviewer
Z = The reported result is based on the combined responses from coeluting compounds
* = Data outside of control limits.

13.6 Data Management and Records: All electronic and hardcopy data is managed, retained, and archived as specified in laboratory SOP LP-QA-0014 *Laboratory Records*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009 *Method Detection Limits.*

14.2 A demonstration of analyst capability (IDOC) is required before use of this SOP and any time there is a significant change in instrument type, personnel or test method.

14.3 Employee Training, and IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

14.4 The laboratory statistically derived control limits used to evaluate accuracy, precision and surrogate recoveries are provided in Table 2. The control limits for accuracy are based on compiled data and are set at 3 standard deviations around the mean using the procedures described in laboratory SOP LP-QA-012 *Control Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 The following waste streams are produced when this method is carried out:

- Vials containing sample extracts

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with

Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (Method 8270C), Revision 3, December 1996, USEPA SW-846 Methods for Evaluating Solid Waste, Update III.

17.0 SOP REVISION HISTORY

The following changes were made in this revision:

Section 5:	Updated Safety section.
Section 7:	7.1 - Removed solvents not used in analytical method.
Section 10:	10.3 - Added additional quantification options. 10.4 - 10.6 Added detail about repeating CCV. 10.7 Added Troubleshooting.
Section 12:	Moved calculations to Appendix A. Moved data reporting to Section 13.
Section 13:	13.2 - Added detail regarding the use of DoD LCS and Surrogate Limits. 13.3 – Added list of poorly performing analytes. 13.4 - Added SOP reference for Data Management & Records. 13.5 – Added data flags.
Section 14:	Completely revised section.
Section 17:	New Section added.
Table 1A &B:	Added Footnotes. Eliminated Standard criteria. Added internal standard assignment.
Table 2:	Revised material list to solvents used in analysis only.
Table 3:	Changed DDT Breakdown Criteria from < 20% to ≤20%. Added phthalates as common laboratory contaminant.
Table 4:	Combined Table 4, 5, and 6, updated control limits, added Low Level Water and Soil Control Limits, added footnotes.
Table 5:	Eliminated original Table 5 (IS assignment and incorporated into Table 1.)
Table 5A&B:	Added footnotes, updated control limits, identified poorly performing analytes.
Appendix A:	New Appendix added containing all calculations.

18.0 TABLES, DIAGRAMS, & FLOWCHARTS

Table 1A:	Target Analyte List, RLs, Characteristic Ions, and IS Assignments
Table 1B:	A9 Extended Target Analyte List, RLs, Characteristic Ions, and IS Assignments
Table 2:	Primary Materials Used
Table 3:	QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action
Table 4:	DFTPP Key Ions and Abundance Criteria
Table 5A:	Control Limits as Accuracy (%R) and Precision (RPD)
Table 5B:	Control Limits as Accuracy (%R) and Precision (RPD) for Extended List

Table 1A: Target Analyte List, RLs, Characteristic Ions, and IS Assignments

Analyte	CAS #	Reporting Limit[1]			Characteristic Ions			Internal Standard Assignment
		Water (ug/L)	Low Soil (ug/Kg)	Med Soil (ug/Kg)	Primary Ion	Secondary Ions		
Pyridine	110-86-1	10	330	10000	79	52	NA	1
N-Nitrosodimethylamine	62-75-9	10	330	10000	74	42	NA	1
Benzaldehyde	100-52-7	25	830	25000	77	105	106	1
Aniline	62-53-3	25	830	25000	93	66	65	1
bis(2-Chloroethyl)Ether	111-44-4	10	330	10000	93	95	NA	1
Phenol (CCC)	108-95-2	10	330	10000	94	65	66	1
2-Chlorophenol	95-57-8	10	330	10000	128	64	130	1
1,3-Dichlorobenzene	541-73-1	10	330	10000	146	148	111	1
1,4-Dichlorobenzene (CCC)	106-46-7	10	330	10000	146	148	111	1
1,2-Dichlorobenzene	95-50-1	10	330	10000	146	148	111	1
Benzyl Alcohol	100-51-6	10	330	10000	108	79	77	1
2,2'-oxybis(1-Chloropropane)	108-60-1	10	330	10000	45	121	NA	1
Acetophenone	98-86-2	10	330	10000	105	77	51	1
2-Methylphenol	95-48-7	10	330	10000	107	108	79	1
Hexachloroethane	67-72-1	10	330	10000	117	201	199	1
N-Nitroso-di-n-propylamine (SPCC)	621-64-7	10	330	10000	70	42	130	1
4-Methylphenol	106-44-5	10	330	10000	107	108	79	1
Nitrobenzene	98-95-3	10	330	10000	77	123	65	2
Isophorone	78-59-1	10	330	10000	82	95	138	2
2-Nitrophenol (CCC)	88-75-5	10	330	10000	139	109	65	2
2,4-Dimethylphenol	105-67-9	10	330	10000	122	107	121	2
bis(2-Chloroethoxy)methane	111-91-1	10	330	10000	93	95	123	2
2,4-Dichlorophenol	120-83-2	10	330	10000	162	164	98	2
Benzoic Acid	65-85-0	35	830	25000	105	122	77	2
1,2,4-Trichlorobenzene	120-82-1	10	330	10000	180	182	145	2
Naphthalene	91-20-3	10	330	10000	128	129	NA	2
4-Chloroaniline	106-47-8	10	330**	10000	127	129	NA	2
Hexachlorobutadiene (CCC)	87-68-3	10	330	10000	225	223	227	2
Caprolactum	105-60-2	10	330	10000	113	55	56	2
2-Methylnaphthalene	91-57-6	10	330	10000	115	141	142	2
4-Chloro-3-methylphenol	59-50-7	10	330	10000	107	144	NA	2
Hexachlorocyclopentadiene (SPCC)	77-47-4	10	330**	10000	237	235	272	3
2,4,6-Trichlorophenol	88-06-2	10	330	10000	196	198	200	3
2,4,5-Trichlorophenol	95-95-4	25	830	25000	196	198	97	3

Analyte	CAS #	Reporting Limit[1]			Characteristic Ions			Internal Standard Assignment
		Water (ug/L)	Low Soil (ug/Kg)	Med Soil (ug/Kg)	Primary Ion	Secondary Ions		
2-Chloronaphthalene	91-58-7	10	330	10000	162	127	164	3
1,1'-Biphenyl	92-52-4	10	330	10000	154	153	152	3
2-Nitroaniline	88-74-4	25	830	25000	65	92	138	3
Acenaphthylene	208-96-8	10	330	10000	152	153	NA	3
Dimethylphthalate	131-11-3	10	330	10000	163	194	NA	3
2,6-Dinitrotoluene	606-20-2	10	330	10000	165	89	NA	3
Acenaphthene	83-32-9	10	330	10000	154	153	152	3
3-Nitroaniline	99-09-2	25	830	25000	138	65	92	3
2,4-Dinitrophenol (SPCC)	51-28-5	25	830	25000	187	107	NA	3
Dibenzofuran	132-64-9	10	330	10000	168	139	NA	3
2,4-Dinitrotoluene	121-14-2	10	330	10000	165	89	NA	3
4-Nitrophenol (SPCC)	100-02-7	25	830	25000	109	81	65	3
Fluorene	86-73-7	10	330	10000	166	165	NA	3
Diethylphthalate	84-66-2	10	330	10000	149	177	150	3
4-Chlorophenyl-phenylether	7005-72-3	10	330	10000	204	206	141	3
4-Nitroaniline	100-01-6	25	830	25000	138	92	108	3
4,6-Dinitro-2-methylphenol	534-52-1	25	830	25000	198	121	106	4
Azobenzene	103-33-3	10	830	25000	182	77	NA	4
N-nitrosodiphenylamine[2] (CCC)	86-30-6	10	330	10000	169	168	167	4
4-Bromophenyl-phenylether	101-55-3	10	330	10000	248	250	141	4
Hexachlorobenzene	118-74-1	10	330	10000	284	142	249	4
Atrazine	1912-24-9	10	330	10000	200	173	215	4
Pentachlorophenol (CCC)	87-86-5	25	830	25000	266	268	204	4
Phenanthrene	85-01-8	10	330	10000	178	179	176	4
Anthracene	120-12-7	10	330	10000	178	176	179	4
Carbazole	86-74-8	10	330	10000	167	139	NA	4
Di-n-butylphthalate	84-74-2	10	330	10000	149	150	104	4
Fluoranthene (CCC)	206-44-0	10	330	10000	202	203	101	4
Pyrene	129-00-0	10	330	10000	202	203	101	5
Benzidine	92-87-5	25	830	25000	184	92	185	5
Butylbenzylphthalate	85-68-7	10	330	10000	149	91	206	5
Benzo(a)anthracene	56-55-3	10	330	10000	228	229	226	5
Chrysene	218-01-9	10	330	10000	228	226	229	5
3,3'-Dichlorobenzidine	91-94-1	10	330	10000	252	254	126	5
bis(2-Ethylhexyl)phthalate	117-81-7	10	330	10000	149	167	NA	5
Di-n-octylphthalate (CCC)	117-84-0	10	330	10000	149	NA	NA	6

Analyte	CAS #	Reporting Limit[1]			Characteristic Ions			Internal Standard Assignment
		Water (ug/L)	Low Soil (ug/Kg)	Med Soil (ug/Kg)	Primary Ion	Secondary Ions		
Benzo(b)fluoranthene	205-99-2	10	330	10000	252	253	125	6
Benzo(k)fluoranthene	207-08-9	10	330	10000	252	253	125	6
Benzo(a)pyrene	50-32-8	10	330	10000	252	253	125	6
Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10000	276	138	277	6
Dibenz(a,h)anthracene	53-70-3	10	330	10000	278	139	279	6
Benzo(g,h,i)perylene	191-24-2	10	330	10000	276	138	277	6
Surrogate Compounds:								
2-Fluorophenol	367-12-4	NA	NA	NA	112	64	NA	1
Phenol-d5	4165-62-2	NA	NA	NA	99	71	42	1
2-Chlorophenol-d4	93951-73-6	NA	NA	NA	132	68	134	1
1,2-Dichlorobenzene-d4	2199-69-1	NA	NA	NA	152	150	115	1
Nitrobenzene-d5	4165-60-0	NA	NA	NA	82	54	128	2
2-Fluorobiphenyl	321-60-8	NA	NA	NA	172	171	NA	3
2,4,6-Tribromophenol	118-79-6	NA	NA	NA	330	332	141	4
Terphenyl-d14	98904-43-9	NA	NA	NA	244	122	212	5
Internal Standards:								
1,4-Dichlorobenzene-d4 (IS1)	3855-82-1	NA	NA	NA	152	115	150	NA
Naphthalene-d8 (IS2)	1146-65-2	NA	NA	NA	136	68	NA	NA
Acenaphthene-d10 (IS3)	15067-26-2	NA	NA	NA	164	162	160	NA
Phenanthrene-d10 (IS4)	1517-22-2	NA	NA	NA	188	94	80	NA
Chrysene-d12 (IS5)	1719-03-5	NA	NA	NA	240	120	236	NA
Perylene-d12 (IS6)	1520-96-3	NA	NA	NA	264	260	265	NA

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.

[2] Analyzed as Diphenylamine due to breakdown in the analytical portion of the procedure.

CCC: Calibration Check Compound

SPCC: System Performance Check Compound

Table 1B: A9 Extended Target Analyte List, RLs, Characteristic Ions, and IS Assignments

Analyte	CAS #	Reporting Limit[1]			Characteristic Ions		
		Water (ug/L)	Low Soil (ug/Kg)	Soil (ug/Kg)	Primary Ion	Secondary Ions	
Ethyl methacrylate	97-63-2	10	330	10000	69	99	41
2-Picoline	109-06-8	10	330	10000	93	66	NA
N-Nitrosomethyl-ethylamine	10595-95-6	10	330	10000	88	42	43
Methyl methanesulfonate	66-27-3	10	330	10000	80	79	65
N-Nitrosodiethylamine	55-18-5	10	330	10000	102	57	44
Ethyl methanesulfonate	62-50-0	10	330	10000	79	109	97
Pentachloroethane	76-01-7	10	330	10000	117	167	83
N-Nitrosopyrrolidine	930-55-2	10	330	10000	100	68	42
o-Toluidine	108-44-1	10	330	10000	106	107	NA
N-Nitrosomorpholine	59-89-2	10	330	10000	56	86	116
N-Nitrosopiperidine	100-75-4	10	330	10000	114	42	55
a,a-Dimethylphenethylamine	122-09-8	10	330	10000	58	91	NA
O,O,O-Triethylphosphorothioate	126-68-1	10	330	10000	198	121	97
p-Phenylenediamine	106-50-3	10	330	10000	108	80	53
Isosafrole	120-58-1	10	330	10000	162	104	131
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330	10000	216	214	218
Safrole	94-59-7	10	330	10000	104	103	131
1,4-Naphthoquinone	130-15-4	10	330	10000	158	102	76
m-Dinitrobenzene	99-65-0	10	330	10000	168	50	76
Pentachlorobenzene	608-93-5	10	330	10000	250	248	215
1-Naphthylamine	134-32-7	10	330	10000	143	116	115
2-Naphthylamine	91-59-8	10	330	10000	143	116	115
2,3,4,6-Tetrachlorophenol	58-90-2	10	330	10000	232	230	131
5-Nitro-o-toluidine	99-55-8	10	330	10000	152	106	79
Thionazin	297-97-2	10	330	10000	97	107	143
Sulfotepp	3689-24-5	10	330	10000	322	202	266
Diallate	2303-16-4	10	330	10000	86	234	43
Phorate	298-02-2	10	330	10000	121	260	NA
sym-Trinitrobenzene	99-35-4	10	330	10000	213	74	91
Phenacetin	62-44-2	10	330	10000	108	179	137
Dimethoate	60-51-5	10	330	10000	87	125	93
4-Aminobiphenyl	92-67-1	10	330	10000	169	168	115
Pentachloronitrobenzene	82-68-8	10	330	10000	237	214	142
Pronamide	23950-58-6	10	330	10000	173	175	145
Disulfoton	298-04-4	10	330	10000	97	142	NA

Analyte	CAS #	Reporting Limit[1]			Characteristic Ions		
		Water (ug/L)	Low Soil (ug/Kg)	Soil (ug/Kg)	Primary Ion	Secondary Ions	
Dinoseb	88-85-7	10	330	10000	211	163	147
Methyl parathion	298-00-0	10	330	10000	109	125	263
4-Nitroquinoline-1-oxide	56-57-5	10	330	10000	101	128	174
Ethyl parathion	56-38-2	10	330	10000	109	97	291
Methapyrilene	91-80-5	10	330	10000	58	97	71
Isodrin	465-73-6	10	330	10000	193	147	66
Aramite	140-57-8	10	330	10000	185	191	319
p-(Dimethylamino)Azobenzene	60-11-7	10	330	10000	120	225	77
Chlorobenzilate	510-15-6	10	330	10000	251	139	111
Kepone	143-50-0	10	830	10000	272	237	143
3,3'-Dimethylbenzidine	119-93-7	10	330	10000	212	213	106
2-Acetylaminofluorene	53-96-3	10	330	10000	181	223	152
7,12-Dimethylbenz(a)anthracene	57-97-6	10	330	10000	256	241	128
3-Methylcholanthrene	56-49-5	10	330	10000	268	253	126
Ethyl methacrylate	97-63-2	10	330	10000	69	99	41
2-Picoline	109-06-8	10	330	10000	93	66	NA
N-Nitrosomethyl-ethylamine	10595-95-6	10	330	10000	88	42	43
Methyl methanesulfonate	66-27-3	10	330	10000	80	79	65
N-Nitrosodiethylamine	55-18-5	10	330	10000	102	57	44
Ethyl methanesulfonate	62-50-0	10	330	10000	79	109	97

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.

Table 2: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] Always add acid to water to prevent violent reactions.
[2] Exposure limit refers to the OSHA regulatory exposure limit.

Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Minimum Frequency	Acceptance Criteria	Recommended Corrective Action[1]
DFTPP	Before initial and continuing calibration, every 12 hours	See Table 4	Reshoot, retune mass spectrometer
DDT Breakdown Check	Daily prior to sample analysis (DoD Only)	Degradation $\leq$20%	Correct problem and repeat check; no samples may be analyzed until acceptance criterion is met.
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CCCs: %RSD $\leq$ 30% SPCCs: mean RF $\geq$ 0.050. Linear Regression: r $\geq$ 0.99 (0.995 for DoD) Quadratic: $r^2 \geq 0.99$	Correct problem and repeat calibration.
ICV	After each initial calibration	%Difference ± 25%	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Beginning of each 12-hour window, as established by a compliant DFTPP.	SPCCs: mean RF $\geq$ 0.050. CCCs: %D $\leq$ 20%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs at different concentrations pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	< RL DoD: $\leq$ ½ RL for all analytes except < RL for phthalates for any sample $\geq$ RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	Evaluated against control limits in Table 5, 4 Marginal Exceedances allowed.	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD	One per extraction batch of 20 or less samples.	Evaluated against control limits in Table 5	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate Standard	All field and QC samples	Evaluated against control limits in Table 5	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.
Internal Standard	All field and QC samples	Area between 50-200% of area of daily calibration internal standard area	Same as above.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that data quality is known and documented. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

Table 4: DFTPP Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
69	Present
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	>40.0 of mass 198
443	17-23% of mass 442

Table 5A: Control Limits[1] as Accuracy (%R) and Precision[2] (RPD)

Analyte	Water				Solid			
	Lab Limits		DoD Limits		Lab Limits		DoD Limits	
	%R	RPD	%R	RPD	%R	RPD	%R	RPD
Pyridine	10-105(5)	50	10-105(3,5)	50	15-105(5)	50	15-105(3,5)	50
N-Nitrosodimethylamine	45-110	30	25-110	30	55-125	30	20-115	30
Benzaldehyde	60-140(6)	30	60-140(6)	30	60-140(6)	30	60-140(6)	30
Aniline	10-110	30	10-110(3)	30	10-105(5)	50	10-105(3,5)	50
bis(2-Chloroethyl)Ether	70-140	30	35-110	30	60-125	30	40-105	30
Phenol	25-90(4)	30	25-90(3,4)	30	**60-140**	30	40-100	30
2-Chlorophenol	**65-140**	30	65-135	30	60-125	30	45-105	30
1,3-Dichlorobenzene	50-125	30	30-100	30	**50-115**	30	40-100	30
1,4-Dichlorobenzene	55-125	30	30-100	30	55-120	30	35-105	30
1,2-Dichlorobenzene	55-130	30	35-100	30	55-120	30	45-95	30
Benzyl Alcohol	**45-150**	30	30-110	30	50-155	30	20-125	30
2,2'-oxybis(1-Chloropropane)	60-150	30	25-130	30	60-130	30	20-115	30
Acetophenone	60-140(6)	30	60-140(6)	30	60-140(6)	30	60-140(6)	30
2-Methylphenol	**55-130**	30	40-110	30	**50-135**	30	40-105	30
Hexachloroethane	**50-140**	30	30-95	30	55-120	30	35-110	30
N-Nitroso-di-n-propylamine	60-130	30	35-130	30	**45-125**	30	40-115	30
4-Methylphenol	**35-130**	30	30-110	30	**35-140**	30	40-105	30
Nitrobenzene	**60-135**	30	45-110	30	55-120	30	40-115	30
Isophorone	**45-135**	30	50-110	30	50-115	30	45-110	30
2-Nitrophenol	70-145	30	40-115	30	**55-135**	30	40-110	30
2,4-Dimethylphenol	**30-165**	30	30-110	30	**25-150**	30	30-105	30
bis(2-Chloroethoxy)methane	**45-160**	30	45-105	30	**55-125**	30	45-110	30
2,4-Dichlorophenol	55-150	30	50-105	30	**55-130**	30	45-110	30
Benzoic Acid	10-70(4)	50	10-70(3,4)	50	25-145(3,4)	50	25-145(3,4)	50
1,2,4-Trichlorobenzene	60-130	30	35-105	30	55-120	30	45-110	30
Naphthalene	**65-135**	30	40-100	30	55-120	30	40-105	30
4-Chloroaniline	**10-95**(5)	30	15-110	30	10-90(3,4)	50	10-90(3,4)	50
Hexachlorobutadiene	**35-140**	30	25-105	30	**50-130**	30	40-115	30
Caprolactum	60-140(6)	30	60-140(6)	30	60-140(6)	30	60-140(6)	30
2-Methylnaphthalene	50-145	30	45-105	30	**45-150**	30	45-105	30
4-Chloro-3-Methylphenol	**50-155**	30	45-110	30	**60-140**	30	45-115	30
Hexachlorocyclopentadiene	10-155(5)	50	10-155(3,5)	50	30-105(3)	40	30-105(3)	40
2,4,6-Trichlorophenol	**55-155**	30	45-110	30	**60-135**	30	45-110	30
2,4,5-Trichlorophenol	**60-140**	30	60-120	30	**60-130**	30	50-110	30
2-Chloronaphthalene	**50-125**	30	50-105	30	**45-120**	30	45-105	30
1,1'-Biphenyl	60-140(6)	30	60-140(6)	30	60-140(6)	30	60-140(6)	30
2-Nitroaniline	**65-140**	30	65-130	30	50-125	30	45-120	30
Acenaphthylene	**60-125**	30	50-105	30	**50-115**	30	45-105	30

Analyte	Water				Solid			
	Lab Limits		DoD Limits		Lab Limits		DoD Limits	
	%R	RPD	%R	RPD	%R	RPD	%R	RPD
Dimethylphthalate	65-140	30	25-125	30	55-120	30	50-110	30
2,6-Dinitrotoluene	**70-135**	30	50-115	30	**55-130**	30	50-110	30
Acenaphthene	**60-135**	30	45-110	30	55-120	30	45-110	30
3-Nitroaniline	30-95	30	20-125	30	**20-85**	30	25-110	30
2,4-Dinitrophenol	35-150	30	15-140	30	**25-160**	30	15-130	30
Dibenzofuran	**65-140**	30	55-105	30	**55-120**	30	50-105	30
2,4-Dinitrotoluene	60-130	30	50-120	30	**50-120**	30	50-115	30
4-Nitrophenol	10-110[4]	30	10-110[3,4]	30	30-155	30	15-140	30
Fluorene	**65-135**	30	50-110	30	**50-125**	30	50-110	30
Diethylphthalate	**55-140**	30	40-120	30	**50-125**	30	50-115	30
4-Chlorophenyl-phenylether	**60-140**	30	50-110	30	**50-120**	30	45-110	30
4-Nitroaniline	50-135	30	35-120	30	**25-115**	30	35-115	30
4,6-Dinitro-2-methylphenol	**55-180**	30	40-130	30	55-160	30	30-135	30
N-nitrosodiphenylamine	65-125	30	50-110	30	**45-130**	30	50-115	30
Azobenzene	65-135[3]	35	65-135[3]	35	60-140	30	60-140[6]	30
4-Bromophenyl-phenylether	**55-150**	30	50-115	30	**55-130**	30	45-115	30
Hexachlorobenzene	**55-150**	30	50-110	30	55-125	30	45-120	30
Atrazine	60-140[6]	30	60-140[6]	30	60-140[6]	30	60-140[6]	30
Pentachlorophenol	**50-165**	30	40-115	30	50-140	30	25-120	30
Phenanthrene	**70-135**	30	50-115	30	**60-125**	30	50-110	30
Anthracene	**70-135**	30	55-110	30	**60-125**	30	55-105	30
Carbazole	**60-140**	30	50-115	30	55-125	30	45-115	30
Di-n-butylphthalate	**60-135**	30	55-115	30	**50-120**	30	55-110	30
Fluoranthene	50-140	30	55-115	30	**50-120**	30	55-115	30
Pyrene	**70-160**	30	50-130	30	**35-175**	30	45-125	30
Benzidine	10-235[5]	50	10-235[3,5]	50	10-120[5]	50	10-120[3,5]	50
Butylbenzylphthalate	75-140	30	45-115	30	65-145	30	50-125	30
Benzo(a)anthracene	**70-135**	30	55-110	30	**55-130**	30	50-110	30
Chrysene	65-130	30	45-120	30	**60-125**	30	55-110	30
3,3'-Dichlorobenzidine	**10-140**	30	20-110	30	10-120[4]	50	10-120[3,4]	50
bis(2-Ethylhexyl)phthalate	80-145	30	40-125	30	**55-140**	30	45-125	30
Di-n-octylphthalate	70-135	30	35-135	30	60-135	30	40-130	30
Benzo(b)fluoranthene	**40-150**	30	45-120	30	**45-130**	30	45-115	30
Benzo(k)fluoranthene	60-140	30	45-125	30	60-125	30	45-125	30
Benzo(a)pyrene	**65-130**	30	55-110	30	55-120	30	50-110	30
Indeno(1,2,3-cd)pyrene	**45-150**	30	45-125	30	55-135	30	40-120	30
Dibenz(a,h)anthracene	**45-150**	30	40-125	30	35-145	30	40-125	30
Benzo(g,h,i)perylene	**50-140**	30	40-125	30	**45-135**	30	40-125	30
Surrogates								
2-Fluorophenol	30-95	NA	20-110	NA	45-110	NA	35-105	NA

Analyte	Water				Solid			
	Lab Limits		DoD Limits		Lab Limits		DoD Limits	
	%R	RPD	%R	RPD	%R	RPD	%R	RPD
Phenol-d5	10-75[4]	NA	10-75 [3,4]	NA	**50-120**	NA	40-100	NA
2-Chlorophenol-d4	55-120	NA	55-120[3]	NA	50-115	NA	50-115[3]	NA
1,2-Dichlorobenzene-d4	55-120	NA	55-120[3]	NA	50-115	NA	50-115[3]	NA
Nitrobenzene-d5	55-125	NA	40-110	NA	50-115	NA	35-100	NA
2-Fluorobiphenyl	30-95	NA	50-110	NA	**45-110**	NA	45-105	NA
2,4,6-Tribromophenol	50-135	NA	40-125	NA	50-120	NA	35-125	NA
Terphenyl-d14	**55-145**	NA	50-135	NA	**40-145**	NA	30-125	NA

[1] The in-house statistical control limits posted in this table are those in effect on the revision date of this SOP. These limits are subject to change based on performance trends.
[2] RPD for MS/MSD only.
[3] DoD Limit not available so limit is statistically derived laboratory limit.
[4] Identified as poorly performing analyte by DoD. Decisions regarding batch acceptability are not based on analyte in the specific matrix indicated.
[5] Identified as poorly performing analyte by laboratory. Decisions regarding batch acceptability are not based on analyte in the specific matrix indicated.
[6] Default limit.
Note: Where in house limits are outside of DoD limits, this is indicated in bold.

Table 5B: Control Limits[1,2,4] as Accuracy (%R) and Precision[3] (RPD) for Extended List

Analyte	CAS #	Water		Soil	
		%R	RPD	%R	RPD
Ethyl methacrylate	97-63-2	60-140	30	60-140	30
2-Picoline	109-06-8	60-140	30	60-140	30
N-Nitrosomethyl-ethylamine	10595-95-6	60-140	30	60-140	30
Methyl methanesulfonate	66-27-3	60-140	30	60-140	30
N-Nitrosodiethylamine	55-18-5	60-140	30	60-140	30
Ethyl methanesulfonate	62-50-0	60-140	30	60-140	30
Pentachloroethane	76-01-7	60-140	30	60-140	30
N-Nitrosopyrrolidine	930-55-2	60-140	30	60-140	30
o-Toluidine	108-44-1	60-140	30	60-140	30
N-Nitrosomorpholine	59-89-2	60-140	30	60-140	30
N-Nitrosopiperidine	100-75-4	60-140	30	60-140	30
a,a-Dimethylphenethylamine	122-09-8	60-140	30	60-140	30
O,O,O-Triethylphosphorothioate	126-68-1	60-140	30	60-140	30
p-Phenylenediamine	106-50-3	60-140	30	60-140	30
Isosafrole	120-58-1	60-140	30	60-140	30
1,2,4,5-Tetrachlorobenzene	95-94-3	60-140	30	60-140	30
Safrole	94-59-7	60-140	30	60-140	30
1,4-Naphthoquinone	130-15-4	60-140	30	60-140	30
m-Dinitrobenzene	99-65-0	60-140	30	60-140	30
Pentachlorobenzene	608-93-5	60-140	30	60-140	30
1-Naphthylamine	134-32-7	60-140	30	60-140	30
2-Naphthylamine	91-59-8	60-140	30	60-140	30
2,3,4,6-Tetrachlorophenol	58-90-2	60-140	30	60-140	30
5-Nitro-o-toluidine	99-55-8	60-140	30	60-140	30
Thionazin	297-97-2	60-140	30	60-140	30
Sulfotepp	3689-24-5	60-140	30	60-140	30
Diallate	2303-16-4	60-140	30	60-140	30
Phorate	298-02-2	60-140	30	60-140	30
sym-Trinitrobenzene	99-35-4	60-140	30	60-140	30
Phenacetin	62-44-2	60-140	30	60-140	30
Dimethoate	60-51-5	60-140	30	60-140	30
4-Aminobiphenyl	92-67-1	60-140	30	60-140	30
Pentachloronitrobenzene	82-68-8	60-140	30	60-140	30
Pronamide	23950-58-6	60-140	30	60-140	30
Disulfoton	298-04-4	60-140	30	60-140	30
Dinoseb	88-85-7	60-140	30	60-140	30
Methyl parathion	298-00-0	60-140	30	60-140	30
4-Nitroquinoline-1-oxide	56-57-5	60-140	30	60-140	30
Ethyl parathion	56-38-2	60-140	30	60-140	30

Analyte	CAS #	Water		Soil	
		%R	RPD	%R	RPD
Methapyrilene	91-80-5	60-140	30	60-140	30
Isodrin	465-73-6	60-140	30	60-140	30
Aramite	140-57-8	60-140	30	60-140	30
p-(Dimethylamino)Azobenzene	60-11-7	60-140	30	60-140	30
Chlorobenzilate	510-15-6	60-140	30	60-140	30
Kepone	143-50-0	60-140	30	60-140	30
3,3'-Dimethylbenzidine	119-93-7	60-140	30	60-140	30
2-Acetylaminofluorene	53-96-3	60-140	30	60-140	30
7,12-Dimethylbenz(a)anthracene	57-97-6	60-140	30	60-140	30
3-Methylcholanthrene	56-49-5	60-140	30	60-140	30

[1] The in-house statistical control limits posted in this table are those in effect on the revision date of this SOP. These limits are subject to change based on performance trends.
[2] Limits are default.
[3] RPD for MS/MSD only.

Appendix A: Equations

Response Factor (RFx) = $\frac{\text{Area}_x \text{ X Concentration}_{is}}{\text{Area}_{is} \text{ X Concentration}_x}$

Where: x=compound, is = Internal Standard

Relative Retention Time (RRT) = $\frac{\text{Retention Time}_x}{\text{Retention Time}_{is}}$

where: x=compound, is = Internal Standard

Mean Response Factor ($\overline{RF}$) = $\frac{\sum_{i=1}^{n} RF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Response Factor (SD) = $\sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Response = $\frac{SD}{\overline{RF}} \times 100\%$

Percent Difference (%D) = $\frac{RF_v - \overline{RF}}{\overline{RF}} \times 100\%$

where: RF_v = Response Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $\frac{\text{Calculated Concentration – Theoretical Concentration}}{\text{Theoretical Concentration}}$ X 100%

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD) = $\frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration (for average RF quantification)

Aqueous Samples

$$C_x = \frac{A_x \times C_{IS} \times V_t}{A_{IS} \times MeanRF \times V_o \times V_i} \times DF$$

Where:

C_x	=	Concentration of compound (ug/L)
A_x	=	Area of quantitation ion
C_{IS}	=	Concentration of associated internal standard (ng)
V_t	=	Extract Volume (uL)
A_{IS}	=	Area of quantitation ion for associated internal standard.
Mean RF	=	Mean Response Factor from initial calibration, or 1 for a TIC or Alkane
V_o	=	Sample volume (mL)
V_I	=	Volume injected (uL)
DF	=	Dilution Factor.

Solid Samples

$$C_x = \frac{A_x \times C_{IS} \times V_t \times GPC \times 10^3 g/Kg}{A_{IS} \times MeanRF \times W_s \times S \times V_i \times 10^3 ng/ug} \times DF$$

Where:

C_x	=	Concentration of compound (ug/Kg)
A_x	=	Area of quantitation ion for compound.
A_{IS}	=	Area of quantitation ion for associated internal standard.
C_{IS}	=	Concentration of associated internal standard (ng)
V_t	=	Volume of final extract (uL)
GPC	=	GPC Factor
Mean RF	=	Mean Response Factor from initial calibration, or 1 for a TIC or Alkane
W_s	=	Weight of sample (g)
S	=	Percent Solids (as a decimal)
V_i	=	Volume injected (uL)
DF	=	Dilution Factor

Sample Concentration (for linear regression or quadratic equation quantification)

Aqueous Samples

$$C_x = \frac{Ce \times V_t}{V_o \times V_i} \times DF$$

Where:

C_x	=	Concentration of compound (ug/L)
Ce	=	ng analyte from the curve
V_t	=	Extract Volume (uL)
V_o	=	Sample volume (mL)
V_i	=	Volume injected (uL)
DF	=	Dilution Factor.

Solid Samples

$$C_x = \frac{Ce \times V_t \times GPC \times 10^3 g/Kg}{W_s \times S \times V_i \times 10^3 ng/ug} \times DF$$

Where:

C_x	=	Concentration of compound (ug/Kg)
Ce	=	ng analyte from the curve
GPC	=	GPC Factor
W_s	=	Weight of sample (g)
S	=	Percent Solids (as a decimal)
V_i	=	Volume injected (uL)
DF	=	Dilution Factor

Appendix B: Terms & Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed.

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Calibration Check Compounds (CCCs): Selective analytes from the compound list that are used 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.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Internal Standard: a known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery of the for each analyte.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

System Performance Check Compounds (SPCCs): Selective analytes from the compound list that are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system.

STL BURLINGTON
SOP CHANGE-IN-PROGRESS ATTACHMENT (CIPA)

SOP Title: ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY SW-846 METHOD 8081A & EPA TO-4A
SOP No: LM-GC-8081
Revision: **7**
Date Effective: 12/12/05
CIPA Date Effective: 07/27/06

Approval Signatures:

QA Manager: Kirstin McCracken **Date:** July 27, 2006

Kirstin McCracken

Organic Manager: Jacob L. Bailey **Date:** July 27, 2006

Jacob L. Bailey

The following revisions or additions in BOLD TEXT were made to the referenced SOP. These changes were implemented on the CIPA Date Effective indicated above.

Page 7: Section 10.5 ICV – Second Source Standard

After each calibration, verify the accuracy of the initial calibration by analyzing the ICV (Appendix A). The calculated concentration of each analyte must be within ± ~~**15%**~~ **20%** of the theoretical concentration. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate. The acceptance criteria must be met on both columns for single component pesticides.

Page 16: Revise Table 4: The revisions are shown on the next page.

SOP CHANGE-IN-PROGRESS ATTACHMENT (CIPA)
SOP No: LM-GC-8081 r7

Table 4: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[1]
Breakdown Check	Before initial calibration and each CCV.	Degradation ≤15% for Endrin and DDT each	Correct problem, reanalyze, repeat calibration.
ICB	After Column Prime	< RL DoD: ≤ ½ RL	Continue to run ICB until acceptance criteria are met.
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CF: RSD ≤ 20% Linear Regression: r ≥ 0.99 (0.995 for DoD) Quadratic: r^2 ≥ 0.99	Correct problem and repeat initial calibration.
ICV	After each initial calibration	%Difference ± ~~15%~~ **20%** from expected value for each analyte	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±15%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs at different concentrations pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and bracketed samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	< RL DoD: ≤ ½ RL If analyte in any sample ≥ RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	Evaluated against control limits in Table 2, 1 Marginal Exceedance allowed.	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch SD: Per client request	Evaluated against control limits in Table 1	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate Spike	All field and QC samples	Evaluated against control limits in Table 2	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that data quality is known and documented. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

STANDARD OPERATING PROCEDURE
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY
SW-846 METHOD 8081A & EPA TO- 4A
Applicable Matrices: Non-Potable Water, Solid & Chemical Materials, Tissue and Air

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APPROVAL SIGNATURES

Laboratory Director: Christopher A. Ouellette — Date: December 12, 2005

QA Manager: Kirstin L. McCracken — Date: December 12, 2005

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1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the determination of concentrations of organochlorine pesticides in extracts derived from non-potable water, solids, tissue, air, and chemical materials including TCLP leachates, using dual column Gas Chromatography with Electron Capture Detectors (GC/ECD). This SOP is applicable to the analytical procedure only; the extraction and extract cleanup methods referenced in this SOP are described in the following laboratory SOPs:

LM-OP-3510	Separatory Funnel Extraction
LM-OP-3540	Soxhlet Extraction
LM-OP-3541	Automated Soxhlet Extraction
LM-OP-3550	Ultrasonic Extraction
LM-OP-Cleanup	Extract Cleanup Procedures
LM-OP-GPC	Gel Permeation Chromatography (GPC)

1.2 The analytes that can be determined by this procedure and their associated Reporting Limits (RL) are listed in Table 1, Section 18.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample is extracted using an appropriate matrix-specific extraction technique. After extraction, the extract may be subject to cleanup depending on the nature of sample matrix and the target analytes. After cleanup, the extract is analyzed by injecting a 2-µl aliquot into a dual capillary column GC/ECD.

2.2 This procedure is based on SW-846 Method 8081A, Organochlorine Pesticides by Gas Chromatography and Compendium Method TO-4A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling followed by GC/MD (GC/Multi-detector).

3.0 DEFINITIONS

A list of terms and definitions is given in Appendix C.

4.0 INTERFERENCES

4.1 Contaminated solvents, reagents or equipment can cause interferences. To reduce the occurrence of this type of interference, glassware must be cleaned thoroughly before use following the procedure given in laboratory SOP LM-OP-Glass *Glassware Washing*. Solvents and acids are lot- tested and approved for use before delivery to the laboratory in accordance with STL-T-001 *Testing of Solvent and Acids*. Solvents should be stored in an area away from organochlorine compounds to minimize contamination.

4.2 Phthalate esters introduced during sample preparation can pose a problem in the determination of pesticides. Common flexible plastics contain varying amounts of phthalate esters, and these can be easily extracted or leached during extraction. To minimize this interference, avoid contact with any plastic materials.

4.3 The presence of PCBs may interfere with the analysis of organochlorine pesticides. This interference is most severe for multi-component analytes such as Chlordane and Toxaphene.

4.4 Non-target compounds co-extracted from the sample matrix can also cause interference, the extent of which will vary considerably depending on the nature of the samples. Elemental sulfur is often found in sediment samples and its presence will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Samples are screened before analysis and those samples that contain high levels of sulfur are subject to cleanup using activated copper before analysis (SW-846 3660B). Waxes, lipids, other high molecular weight materials and co-eluting organophosphorous pesticides may be removed by extract cleanup with GPC (SW-846-3640A). Co-eluting chlorophenols can be eliminated by cleanup with silica gel (SW-846 3630C), or Florisil (SW-846 3620B).

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 Specific Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature before working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 Primary Materials Used

Table 3, Section 18 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **Note: The table does not include all materials used in the procedure. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used can be found in Section 7. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 EQUIPMENT AND SUPPLIES

6.1 Autosampler Vials, National Scientific or equivalent.

6.2 Computer Hardware/Software: GC Acquisition Platform - VAX 4505 (GVAX) Multichrom V2.11. Data Processing - Hewlett-Packard 9000-series computers, an HP9000 D250 (Chemsvr4) and an HP 9000 K200 (Chemsvr5)/ HP-UX 10.20 and Target V3.5.

6.3 GC/ECD: with dual columns, dual ECDs, and auto-sampler capable of a 2-µl injection split onto two columns: HP 5890s with Leap Technology CTC AS200SE and A200S Fisons auto-samplers, or equivalent.

6.4 GC Columns:

- RTX-CLPesticides (30m x 0.32 mmID x 0.25µm), Restek or equivalent
- RTX-CLPesticides2, (30m x 0.32 mm ID x 0.25µm), Restek or equivalent

6.5 Hydrogen Generator: Whatman.

6.6 Volumetric Syringes, Class “A” (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

- Hexane (JT Baker Ultra-Resi analyzed) or equivalent.

7.2 Standards

Stock standard solutions are purchased made from commercial vendors. Intermediate and working standards solutions are prepared in the laboratory by diluting a known volume of stock standard in an appropriate solvent and diluting to a specified volume. Standard preparation procedures for intermediate and working standard solutions are provided in Appendix A.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 Sample extracts must be stored at 4°C ± 2° until the time of analysis. The analytical holding time is 40 days from date of sample extraction.

8.2 Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 The minimum frequency requirements, acceptance criteria and recommended corrective action for all QC samples are summarized in Section 18, Table 4. Below is a summary of each type of QC sample that is analyzed with the method.

9.2 A Method Blank (MB) and Laboratory Control Sample (LCS) are prepared with each extraction batch. These samples show that the laboratory is in control, independent of the sample matrix.

9.3 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) are prepared with each extraction batch. Project specific MS/MSD are performed per client request. Sample Duplicates (SD) are performed per client request. These samples show the effect of the sample matrix on the accuracy and precision of the method.

9.4 A Surrogate spike is added to all field and QC samples before extraction to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix.

9.5 Instrumental QC standards include a Breakdown Check Standard (BCS) before each Initial Calibration (ICAL), at the beginning of any daily run, and every 10 samples within a sequence. A five-point ICAL is generated for each individual single component pesticide, and each qualitatively identified multi-component pesticide. After the ICAL, an Initial Calibration Verification (ICV) standard, also referred to as a second source standard, is analyzed to verify the ICAL standard formulation. Continuing Calibration Verification (CCV) standards are analyzed before sample analysis, every ten samples thereafter, and at the end of the run to assess instrument drift.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Operating Conditions

Install a 5-m deactivated guard column to the injection port and connect the guard column to the separate analytical columns attached to dual ECD detectors using a glass "Y".

The recommended instrument operating conditions are as follows:

Initial Temperature:	120°C for 1 minute
Temperature Program:	20°C per minute to 220°C to 6°C per minute to 245°C to 9°C per minute to 300°C. Hold for 3 minutes.
Detector Temperature	300°C
Injector Temperature:	200°C
Injection volume:	2-µL
Carrier Gas:	Hydrogen (supplied by hydrogen generator)

Optimize the flow rate of the carrier gas by injecting an un-retained substance onto the column at an isothermal oven state and adjusting the flow to obtain the recommended dead volume time.

The GC conditions listed above may be changed provided the elution orders are documented and compound separations are maintained but once the operating conditions are established, use the same conditions for both calibration and analysis of field samples.

10.2 Initial Calibration

If the instrument has been idle for longer than 8 hours, analyze a Column Prime Standard (Appendix A) before initial calibration. Analyze an Initial Calibration Blank (ICB) after the column prime standard. The instrument blank should be free of any target analytes at $\geq$ RL ($\geq$ 1/2 RL for Department of Defense work (DoD)).

Initial calibration is performed before any sample analysis (initial method set-up), whenever a new column is installed, when significant instrument maintenance has been performed, and when the CCV standard does not meet acceptance criteria. Significant instrument maintenance includes installing new columns, changing the lengths of the analytical columns and baking or installing detectors.

Analyze the BCS containing Endrin and DDT. The individual breakdown values must be $\leq$ 15% (Appendix B). If the breakdown criteria are not met, correct the problem and reanalyze. The BCS must be analyzed before initial calibration, at the start of each sequence, every 10 samples, and at the end of the run.

Prepare the calibration standards as described in Appendix A. Unless otherwise specified for a specific project, the individual pesticides are calibrated using a minimum of five calibration points. The initial calibration for multi-component pesticides employs a single-point standard at the low-point of the calibration range that is used for pattern recognition. When a multi-component pesticide is detected, a five-point curve is established for it, and the extract is re-analyzed.

Inject 2-µl of each calibration standard and calculate the Calibration Factor (CF), mean CF and Percent Relative Standard Deviation (% RSD) for each analyte on both columns (Appendix B). If the %RSD $\leq$ 20%, the calibration is acceptable and the mean CF may be used for quantification. If this criterion is not met, either use another suitable quantification method, or correct the problem and repeat the calibration. The acceptance criteria must be met on both columns, with the exception of Technical Chlordane and Toxaphene, which require pattern recognition rather than second column confirmation.

10.3 Alternate Quantification. In some cases, it may be preferable to use either linear regression or Quadratic Equations to quantify the compounds. The following approaches may be used:

Linear Regression - A curve of concentration vs. peak area is generated for each analyte and the correlation coefficient is calculated. The calibration must have a correlation coefficient (r) $\geq$ 0.99 (0.995 for DoD) for acquisition of samples to continue. The use of linear regression requires a minimum of 5 calibration points. See SW-846 Method 8000B for linear regression calculations.

Quadratic Equation - For some compounds, the response is not linear, and in this case a quadratic equation may be employed. For those compounds, the coefficient of determination (r^2) is generated. The coefficient of determination must be $\geq$ 0.99 for acquisition to continue. The uses of quadratic equations require a minimum of 6

calibration points for second order regression and 7 points for third order regression. See SW-846 Method 8000B for quadratic equation calculations.

Once a method of calibration is chosen for a specific compound, it must be consistent throughout the entire analytical sequence until a new initial calibration is generated.

10.4 Retention Time (RT) Windows

When a new GC column is installed, RT windows are generated by analyzing three standards over a 72-hour period and calculating the mean RT and Standard Deviation (SD). The RT window is calculated as mean RT $\pm$ 3SD of the three standards. If the SD is <0.01 minutes, the laboratory may use a default SD of 0.01 minutes.

If, in the professional judgment of the analyst, this results in an RT window that is too tight and would favor false negatives, the laboratory may opt use an alternate method to determine the RT windows as follows: using the RT of the midpoint initial calibration standard, calculate the RT window using $\pm$ 0.05 minutes from the midpoint of the RT in the initial calibration.

10.5 ICV – Second Source Standard

After each calibration, verify the accuracy of the initial calibration by analyzing the ICV (Appendix A). The calculated concentration of each analyte must be within $\pm$ 15% of the theoretical concentration. If this criterion is not met, correct the problem and reanalyze the ICV. If the reanalysis fails, remake the calibration standards and recalibrate. The acceptance criteria must be met on both columns for single component pesticides.

10.6 Continuing Calibration

A BCS is analyzed at the beginning of each sequence, every 10 samples, and at the end of the run. The BCS is evaluated as per Section 10.2.

A CCV, at or below mid-calibration range, is analyzed each day before sample analysis, after every ten injections and at the end of each analytical batch to monitor instrument drift. The concentration of the CCV is varied. The CCV should include all single component analytes. When a multi-component analyte is detected in a field sample, analyze a multi-component CCV varying the concentration within the analytical batch.

Calculate the CF and percent difference or drift (Appendix B) for each analyte on both columns. The percent difference or drift must be within $\pm$15% for each analyte. Compare the RT of each analyte in the CCV with the RT windows; the RT must be within the window established in 10.4. The acceptance criteria must be met on both columns, with the exception of Technical Chlordane and Toxaphene, which require pattern recognition rather than second column confirmation.

If the CCV fails, it may be repeated once. If it still fails, corrective action must be taken. The sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If the two CCVs do not meet the criteria,

recalibration is required prior to running samples. Samples must be bracketed by passing CCVs, and samples before and after CCV failure must be reanalyzed, unless the CCV is high and there are no detects in the associated samples.

10.7 Troubleshooting: the following items can be checked in case of calibration failures:

- Endrin/DDT Breakdown failure – Perform injection port maintenance (change septum and liner).
- ICAL Failure – Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if chromatography has degraded as evidenced by peak shapes.
- CCV Failure – Perform Injection port maintenance; if injection port maintenance does not restore CCV, install a new guard column.
- Needle crushed during injection - Replace the needle.
- Auto-sampler failure - Reset the auto-sampler.
- Power failure - Reset run in Multichrom and re-acquire or re-initiate run sequence.

11.0 PROCEDURE

11.1 Transfer approximately 100-µl of each QC standard and sample extract to an auto-sampler vial and place the vials in the auto-sampler. Arrange the samples in a sequence that begins with the calibration standards followed by the analysis of QC samples, field samples and CCVs.

11.2 An example analysis sequence that includes initial calibration is given below:

Injection Number	Lab Description	
1	Column Priming Standard	Column Prime
2	Initial Calibration Blank	ICB – repeat until meets criteria in 10.2
3	Breakdown Check Standard	BCS
4	Single Component Level 1	INDAB-1
5	Single Component Level 2	INDAB-2
6	Single Component Level 3	INDAB-3
7	Single Component Level 4	INDAB-4
8	Single Component Level 5	INDAB-5
9	Toxaphene - 500 ppb	TOX500
10	Technical Chlordane - 50 ppb	T.CHLOR50
11	ICV – Second Source Standard	ICV
12-21	10 injections	QC and Field Samples
22	Breakdown Check Standard	BCS
23	Continuing Calibration Verification Standard	CCV
24-33	10 injections	QC and Field Samples
34	Breakdown Check Standard	BCS
35	Continuing Calibration Verification Standard	CCV

Injection Number	Lab Description
Repeat until ending with BCS and CCV	

11.3 Enter the sample ID's into the data acquisition program in the same order that the samples were placed in the auto-sampler. Start the analytical sequence and acquire the data.

11.4 Cleaning blanks (CBLK) consisting of hexane may be analyzed after high-level samples at the discretion of the analyst.

11.5 The data system tentatively identifies target analytes by comparing the retention time of the peaks to the RT windows. Detects are confirmed if the peak is within the retention time window on both columns. The retention time shift of the surrogates in each sample is evaluated and the RT window may be compensated in the direction of the shift to prevent false negatives. If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP LP-LB-0006 *Manual Integration.*

11.6 Quantification of target analytes is performed by the data system using the equations given in Appendix B. The multicomponent pesticides are identified by pattern recognition and are quantified using 3-5 major peaks using the five-point curve (Section 10.2). Calculate a calibration factor for each of the 3-5 major peaks for each calibration level using height or area. Use the average CF to calculate an extract concentration for each of the 3-5 major peaks, and report the average of the results.

11.7 After analysis is complete, evaluate the results against the performance criteria given in Section 10 and Table 3, Section 18 and perform corrective action as necessary.

11.8 Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should ideally result in a determination within the upper half of the calibration curve.

12.0 CALCULATIONS

See Appendix B.

13.0 DATA ASSESSMENT, CORRECTIVE ACTION & REPORTING

13.1 Data Review and Corrective Action

Review the samples, standards and QC samples against the acceptance criteria in Table 4. If the results do not fall within the established limits, perform the recommended corrective action. If corrective action is unsuccessful, document the situation with a nonconformance report and/or qualify the data using an appropriate data qualifier (see Appendix C for data qualifier definitions). For additional guidance regarding the laboratory's protocol and required elements for each level of data review refer to laboratory SOP LP-LB-003 *Data Review*.

In the absence of project specific requirements, use the control limits specified in Table 1. The control limits in Table 1 are based on in-house statistically generated limits. In some cases, the in-house limits were outside of Department of Defense (DoD) limits as specified in the Quality Systems Manual for Environmental Laboratories. Where this is the case, the laboratory uses the stricter, DoD limits that are presented in bold in Table 1. For DoD projects, the in-house laboratory limits are also included in the project report.

Based on the number of analytes in the LCS (21), it is statistically likely that at least one analyte will marginally exceed the control limits; therefore, 1 marginal exceedance is allowed. A marginal exceedence (ME) is defined as being outside of the control limit of $\pm$ 3 SD, but within $\pm$ 4 SD. In order to easily calculate these limits, the following equation may be used:

$$\text{ME (Lower Limit)} = \text{Lower Limit} - \frac{(\text{Upper Limit} - \text{Lower Limit})}{6}$$

$$\text{ME (Upper Limit)} = \text{Upper Limit} + \frac{(\text{Upper Limit} - \text{Lower Limit})}{6}$$

13.2 Data Reporting

Unless otherwise specified; the higher result between the two columns is reported. The Relative Percent Difference (RPD) of the two results is calculated, and if the RPD is greater than 40% it is reported with a data flag. If, in the analyst's judgment, the higher result is due to overlapping peaks, or interference peaks, the lower of the two results should be reported with a data flag, and the issue discussed in the project narrative.

The laboratory's RL for each target analyte is provided in Table 1. Report the data to the RL adjusted for sample matrix, percent moisture, and sample dilution/concentration. The reporting limit is the threshold value below which results are reported as non-detected. Report sample results that have concentrations for a target analytes less than the RL with a "U" qualifier. Unless otherwise specified, report the results for solid matrices on a dry weight basis.

Some projects may require reporting positively identified target analytes less than the RL. In this case, the analyte can be qualitatively detected but not accurately quantified. Flag all results less than the RL with a "J" data qualifier (Appendix C).

Some projects may require RLs that are less than the laboratory's routine RL. Sample results may be reported to the project RL if the project RL is greater than the Quantification Limit (QL) and above the MDL. In this context, the QL is defined as the concentration of the low calibration standard. If the project RL is less than the QL, all values less than the QL must be reported as estimated and qualified with a "J".

Further guidance on the application and use of the MDL, RL, and QL is provided in laboratory SOP LP-LB-009 *Determination of Method Detection Limits.*

Further guidance on the application and use of the MDL, RL, and QL is provided in laboratory SOP LP-LB-009 *Determination of Method Detection Limits.*

13.3 Data Management and Records: All electronic and hardcopy data is managed, retained, and archived as specified in laboratory SOP LP-QA-0014 *Laboratory Records*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009 *Method Detection Limits.*

14.2 A demonstration of analyst capability (IDOC) is required before use of this SOP and any time there is a significant change in instrument type, personnel or test method.

14.3 Employee Training, and IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

14.4 The laboratory statistically derived control limits used to evaluate accuracy, precision and surrogate recoveries are provided in Table 2. The control limits for accuracy are based on compiled data and are set at 3 standard deviations around the mean using the procedures described in laboratory SOP LP-QA-012 *Control Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 Waste Streams Produced by the Method

The following waste stream is produced when this procedure is performed:

- Vials containing sample extracts

Waste streams produced in the laboratory are accumulated in satellite containers located in the work area. The satellite containers are labeled “Hazardous Waste” along with the type of waste category generated. Authorized personnel routinely transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in laboratory SOP LP-LB-001 *Hazardous Waste*.

16.0 REFERENCES

16.1 Organochlorine Pesticides by Gas Chromatography (Method 8081A), Revision 1, December 1996, USEPA SW-846 Methods for Evaluating Solid Waste, Update III.

16.2 Compendium Method TO-4A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling followed by GC/MD, EPA Center for Environmental Research Information, January 1999.

17.0 SOP REVISION HISTORY

The following changes were made in this revision:

Section 6:	Added computer hardware and software and hydrogen generator.
Section 7:	7.1 - Removed solvents not used in analytical method.
Section 10:	10-2 & 10.6 - Clarified that confirmation is not required for multi-component pesticides. 10.3 - Added additional quantification options. 10.4 - Changed RT window requirements to assess RT window from initial calibration curve. 10.6 Added detail about repeating CCV. 10.7 Added Troubleshooting.
Section 13:	13.1 - Added detail regarding the use of DoD LCS and Surrogate Limits. 13.2 – Moved dual column reporting from Section 11. Changed to reporting higher value and flagging at 40%. 13.3 - Added SOP reference for Data Management & Records.
Section 17:	New Section added.
Table 1:	Added Mirex. Added Footnotes.
Table 2:	Updated control limits. Added footnotes and Mirex.
Table 3:	Formerly Table 2. Removed solvents not used in analytical method.
Table 4:	Formerly Table 3 - Corrected DDT/Endrin breakdown limit from <15% to ≤15%, and removed < 30%. Re-worded corrective action for MB, MS/MSD, SD, and Surrogate. Added detail to corrective action section.
Appendix B:	Corrected DDT breakdown calculation. Added percent drift calculation. Corrected Sample concentration, solid calculation.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

Table 1:	Target Analyte List and Reporting Limits
Table 2:	Control Limits as Accuracy (%R) and Precision (RPD)
Table 3:	Primary Materials Used
Table 4:	QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action
Appendix A:	Standard Preparation Tables
Appendix B:	Equations
Appendix C:	Terms and Definitions

Table 1: Target Analyte List and Reporting Limits

Reporting Limit[1]			
Analyte	**Water ug/L**	**Solid ug/Kg**	**Air ug/Media**
alpha-BHC	0.050	1.7	0.05
beta-BHC	0.050	1.7	0.05
delta-BHC	0.050	1.7	0.05
gamma-BHC (Lindane)	0.050	1.7	0.05
Heptachlor	0.050	1.7	0.05
Aldrin	0.050	1.7	0.05
Heptachlor Epoxide	0.050	1.7	0.05
Endosulfan I	0.050	1.7	0.05
Dieldrin	0.10	3.3	0.10
4,4-DDE	0.10	3.3	0.10
Endrin	0.10	3.3	0.10
4,4-DDD	0.10	3.3	0.10
Endosulfan II	0.10	3.3	0.10
Endosulfan sulfate	0.10	3.3	0.10
4,4-DDT	0.10	3.3	0.10
Methoxychlor	0.50	17	0.50
Endrin ketone	0.10	3.3	0.10
Endrin aldehyde	0.10	3.3	0.10
alpha-Chlordane	0.050	1.7	0.05
gamma-Chlordane	0.050	1.7	0.05
Technical Chlordane	0.50	17	0.50
Toxaphene	5.0	170	5.0
Mirex[2]	0.10	3.3	0.10
Surrogates:			
Tetrachloro-m-xylene	NA	NA	NA
Decachlorobiphenyl	NA	NA	NA

[1] Reporting Limits represent those that can be achieved in a blank matrix. Individual reporting limits will vary based upon sample matrix, target analyte concentration, co-extracted interferences, and dry weight of samples.

[2] Performed at client request only.

Table 2: Control Limits[1,2] as Accuracy (%R) and Precision[3] (RPD)

Analyte	Water		Solid, Chemical Material, Tissue		Air[4]
	%R	RPD	%R	RPD	%R
alpha-BHC	65-130	≤ 30	60-120	≤ 30	65-125
beta-BHC	65-130	≤ 30	60-125	≤ 30	65-125
delta-BHC	60-125	≤ 30	55-125	≤ 30	65-125
gamma-BHC (Lindane)	65-130	≤ 30	60-120	≤ 30	65-125
Heptachlor	**45-135**	≤ 30	60-125	≤ 30	65-125
Aldrin	50-125	≤ 30	55-120	≤ 30	65-125
Heptachlor Epoxide	70-135	≤ 30	50-135	≤ 30	65-125
Endosulfan I	**60-120**	≤ 30	50-120	≤ 30	65-125
Dieldrin	70-135	≤ 30	**65-125**	≤ 30	65-125
4,4-DDE	65-130	≤ 30	**70-125**	≤ 30	65-125
Endrin	75-140	≤ 30	**60-135**	≤ 30	65-125
4,4-DDD	70-140	≤ 30	60-145	≤ 30	65-125
Endosulfan II	60-125	≤ 30	50-130	≤ 30	65-125
Endosulfan sulfate	70-135	≤ 30	**60-135**	≤ 30	65-125
4,4-DDT	60-135	≤ 30	60-140	≤ 30	65-125
Methoxychlor	75-140	≤ 30	50-140	≤ 30	65-125
Endrin ketone	**75-125**	≤ 30	**65-135**	≤ 30	65-125
Endrin aldehyde	65-130	≤ 30	**35-145**	≤ 30	65-125
alpha-Chlordane	**65-125**	≤ 30	**65-120**	≤ 30	65-125
gamma-Chlordane	70-130	≤ 30	**65-125**	≤ 30	65-125
Technical Chlordane[5]	70-130	≤ 30	70-130	≤ 30	65-125
Toxaphene[5]	70-130	≤ 30	70-130	≤ 30	65-125
Mirex[4,6]	50-150	≤ 30	50-150	≤ 30	50-150
Surrogates:					
Tetrachloro-m-xylene	60-120	NA	**70-125**	NA	55-120
Decachlorobiphenyl	65-125	NA	55-115	NA	55-115

[1] The in-house statistical control limits posted in this table are those in effect on the revision date of this SOP. These limits are subject to change based on performance trends.
[2] Those limits appearing in bold are limits for which the in-house limit it outside of the DoD required limit, and corrective action is taken based on the DoD limit per Section 13.1.
[3] RPD for MS/MSD only.
[4] Limits are default.
[5] Included in TCLP LCS only.
[6] On client request only.

Table 3: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

[1] Always add acid to water to prevent violent reactions.
[2] Exposure limit refers to the OSHA regulatory exposure limit.

Table 4: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[1]
Breakdown Check	Before initial calibration and each CCV.	Degradation ≤15% for Endrin and DDT each	Correct problem, reanalyze, repeat calibration.
ICB	After Column Prime	< RL DoD: ≤ ½ RL	Continue to run ICB until acceptance criteria are met.
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	CF: RSD ≤ 20% Linear Regression: r ≥ 0.99 (0.995 for DoD) Quadratic: r^2 ≥ 0.99	Correct problem and repeat initial calibration.
ICV	After each initial calibration	%Difference ± 15% from expected value for each analyte	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 samples and at the end of the analytical sequence	% Difference or Drift ±15%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs at different concentrations pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and bracketed samples are non-detects.
MB	One per extraction batch of 20 or fewer samples	< RL DoD: ≤ ½ RL If analyte in any sample ≥ RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer samples	Evaluated against control limits in Table 2, 1 Marginal Exceedance allowed.	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
MS/MSD SD	MS/MSD: Per extraction batch SD: Per client request	Evaluated against control limits in Table 1	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate Spike	All field and QC samples	Evaluated against control limits in Table 2	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that data quality is known and documented. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

Appendix A: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory SOP LP-LB-002 *Standard Preparation* for further guidance on the preparation of standard solutions.

All standards are prepared using volumetric glassware, including Class A volumetric flasks, and Hamilton Syringes. Unless otherwise noted, or unless the expiration date of the parent standard is earlier, an expiration date of 6 months from date of preparation is assigned to all intermediate and working standards. If the expiration date of any of parent standards is earlier, that earlier expiration date is used.

Table Legend:
C_{stock} = Concentration of Parent Standard
V_{stock} = Volume of Parent Standard
V_{std} = Volume of Prepared Standard
C_{std} = Theoretical Concentration of Prepared Standard

INTERMEDIATE STANDARDS – in hexane

Toxaphene (50mg/L)

Stock Standard	C_{stock} (mg/L)	V_{stock} (mL)	V_{std} (mL)	C_{std} (mg/L)
Toxaphene*	1000	1.0	20	50

*Restek, Catalog Number 32005 or equivalent.

Technical Chlordane (10mg/L)

Stock Standard	C_{stock} (mg/L)	V_{stock} (mL)	V_{std} (mL)	C_{std} (mg/L)
Technical Chlordane*	1000	0.400	40	10

*****Restek Catalog Number 32021 or equivalent.

WORKING STANDARDS - in hexane

Column Prime Standard – Varied Concentrations

Stock Standard	C_{stock} (mg/L)	V_{stock} (µl)	V_{std} (mL)	C_{std} (ug/L)
Organochlorine Pesticide Mix AB #2*	8-80	1000	20	400/800
TCX/DCB Working Standard	10	800		400

*Restek Catalog Number 32292 or equivalent

Breakdown Check Standard (BCS) – Varied Concentrations

Stock Standard	C_{stock} (mg/L)	V_{stock} (µL)	V_{std} (mL)	C_{std} (µg/L)
Pesticide Performance Evaluation Mix*	10-250	30	30	10-250

*Ultra Catalog Number

ICV – Second Source Standard – Varied Concentrations

Stock Standard	C_{stock} (mg/L)	V_{stock} (µl)	V_{std} (mL)	C_{std} (ug/L)
Organochlorine Pesticide Mix AB # 2*	8-80	100	40	20-200
TCX/DCB Working Standard	10	80		20

*Second source

Mirex (10mg/L)

Stock Standard	C_{stock} (mg/L)	V_{stock} (mL)	V_{std} (mL)	C_{std} (mg/L)
Mirex *	100	1.0	10	10

*Ultra Scientific Catalog Number: PST-720S

TCX/DCB (10mg/L)

Stock Standard	C_{stock} (mg/L)	V_{stock} (mL)	V_{std} (mL)	C_{std} (mg/L)
Pest Surrogate Mix *	200	1.0	20	10

*Restek Catalog Number 32000 or equivalent

CALIBRATION STANDARDS – in hexane

INDAB-5: Pesticide Calibration Level 5 – Varied Concentrations

Stock Standard	C_{stock} (mg/L)	V_{stock} (mL)	V_{std} (mL)	C_{std} (µg/L)
Organochlorine Pesticide Mix AB # 2*	8-80	1.0	100	80-800
Mirex Working Standard	10	1.6		160
TCX/DCB Working Standard	10	0.800		80

*Restek Catalog Number 32292 or equivalent

Prepare Pesticide Calibration Standards Level 1-4 in hexane using INDAB-5 Pesticide Calibration Level 5 as stock:

Pesticide Calibration Curve

INDAB-5 (Calibration Level 5)	INDAB-4	INDAB-3	INDAB-2	INDAB-1
V_{stock} (mL)	40	20	10	2.5
V_{std} (mL)	80	80	80	40

Toxaphene Working Standard - Calibration Level 5

Stock Standard	C_{stock} (mg/L)	V_{stock} (mL)	V_{std} (mL)	C_{std} (µg/L)
Toxaphene Intermediate Standard	50	16	100	8000
TCX/DCB Working Standard	10	0.80		80

Prepare the Toxaphene Calibration Standards Level 1-4 using Toxaphene Calibration Level 5 as the parent component and the volumes given in the following table:

Toxaphene Curve

Toxaphene - CAL Level 5	Tox4000 (µg/L)	Tox2000 (µg/L)	Tox1000 (µg/L)	Tox500 (µg/L)
Vstock (mL)	20	10	5.0	2.5
Vspike (mL)	40	40	40	40

Technical Chlordane Working Standard - Calibration Level 5

Stock Standard	C_{stock} (mg/L)	V_{stock} (mL)	V_{std} (mL)	C_{std} (µg/L)
Technical Chlordane Intermediate	10	8.0	100	800
TCX/DCB Working Standard	10	0.800		80

Prepare the Technical Chlordane Calibration Standards Level 1-4 using Technical Chlordane Calibration Level 5 as the parent component and the volumes given in the following table:

Technical Chlordane Curve

Technical Chlordane (Level 5)	T. Chlor 400 (µg/L)	T. Chlor 200 (µg/L)	T. Chlor 100 (µg/L)	T. Chlor 50 (µg/L)
Vstock (mL)	20	10	5.0	2.5
Vstd (mL)	40	40	40	40

Final Concentration of Organochlorine Pesticides Curve

Analyte*	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)
TCX (surrogate)	5	10	20	40	80
DCB (surrogate)	5	10	20	40	80
Alpha-BHC	5	10	20	40	80
Beta-BHC	5	10	20	40	80
Delta-BHC	5	10	20	40	80
Gamma-BHC	5	10	20	40	80
Heptachlor	5	10	20	40	80
Aldrin	5	10	20	40	80
Heptachlor Epoxide	5	10	20	40	80
Endosulfan I	5	10	20	40	80
Dieldrin	10	20	40	80	160
4-4'-DDE	10	20	40	80	160
Endrin	10	20	40	80	160
Endosulfan II	10	20	40	80	160
4-4'-DDD	10	20	40	80	160
Endosulfan Sulfate	10	20	40	80	160
4,4'-DDT	10	20	40	80	160
Methoxychlor	50	100	200	400	800
Endrin Ketone	10	20	40	80	160
Endrin Aldehyde	10	20	40	80	160
gamma-chlordane	5	10	20	40	80
alpha-chlordane	5	10	20	40	80
Technical Chlordane	50	100	200	400	800
Toxaphene	500	1000	2000	4000	8000

APPENDIX B: CALCULATIONS

% DDT Breakdown = $\frac{\text{Sum of peak area or height (DDE+DDD)}}{\text{Sum of peak area or height (DDE+DDD+DDT)}}$ x 100%

% Endrin Breakdown = $\frac{\text{Sum of peak area or height (Endrin aldehyde + Endrin ketone)}}{\text{Sum of peak area or height (Endrin aldehyde+Endrin ketone+Endrin)}}$ x 100%

Calibration Factor (CF_x) = $\frac{\text{Peak area or height}_{(x)}}{\text{Standard concentration}_{(ug/L)}}$

Mean Calibration Factor ($\overline{CF}$) = $\frac{\sum_{i=1}^{n} CF_i}{n}$

where: n = number of calibration levels

Standard Deviation of the Calibration Factor (SD) = $\sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)2}{n-1}}$

where: n = number of calibration levels

Percent Relative Standard Deviation (RSD) of the Calibration Factor = $\frac{SD}{\overline{CF}} \times 100\%$

Percent Difference (%D) = $\frac{CF_v - \overline{CF}}{\overline{CF}} \times 100\%$

where: CF_v = Calibration Factor from the Continuing Calibration Verification (CCV)

Percent Drift = $\frac{\text{Calculated Concentration – Theoretical Concentration}}{\text{Theoretical Concentration}}$ X 100%

Percent Recovery (%R) = $\frac{C_s}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Field or QC Sample
C_n = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD = $\frac{C_s - C_u}{C_n} \times 100\%$

where: C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

Relative Percent Difference (%RPD) = $\frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$

where: C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

Sample Concentration

Extract

$$C_{extract}\,(ug/L) = \frac{\text{Peak Area (or Height)}}{\overline{CF}}$$

Water

$$C_{sample}\,(ug/L) = C_{extract}\,(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample volume (L)}} \times DF$$

Solids

$$C_{sample}\,(ug/Kg) = C_{extract}\,(ug/L) \times \frac{\text{extract volume (L)}}{\text{sample weight (Kg)}} \times \frac{100}{\%\text{ solids}} \times DF$$

where: DF = Extract Dilution Factor. If no dilution was made, DF=1.

Appendix C: Terms & Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

The qualifiers that are routinely used for this test method are:

U: Compound analyzed for but not detected at a concentration above the reporting limit.
J: Estimated Value
P: There is greater 40% difference for detected concentrations between two GC columns
C: Positive result whose identification has been confirmed by GC/MS
B: Compound is found in the sample and the associated method blank.
E: Compound whose concentration exceeds the upper limit of the calibration range.
D: Concentration identified from a dilution analysis.

X,Y,Z: Laboratory defined flags that may be used alone or combined as needed. If used, provide a description of the flag in the project narrative.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

SOP CHANGE -IN-PROGRESS ATTACHMENT (CIPA)

SOP Title: Separatory Funnel Liquid-Liquid Extraction
SOP No: LM-OP-3510
Revision: 4
Date Effective: 12/12/05
CIPA Date Effective: 02/27/06

QA Manager: Kirstin McCracken **Date:** February 27, 2006

Organic Manager: Jennifer L. Clements **Date:** February 27, 2006

The following revisions or additions in BOLD TEXT were made to the referenced SOP. These changes were implemented on the CIPA Date Effective indicated above.

11.3.3 Nitrogen Blow Down

Use nitrogen blow down when concentration using the K-D technique may be difficult due to high extract viscosity. Place the concentrator tube in a warm bath (**~~65~~ 35**°C) and apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

STANDARD OPERATING PROCEDURE
SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION
SW-846 3510C
Applicable Matrix: Water

APPROVAL SIGNATURES

Laboratory Director: _____ Date: December 12, 2005
Christopher A. Ouellette

QA Manager: _____ Date: December 12, 2005
Kirstin L. McCracken

Organics Manager: _____ Date: December 12, 2005
Jennifer L. Clements

Proprietary Information Statement:

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1.0 SCOPE AND APPLICATION

1.1. This SOP describes the laboratory procedure for the isolation of organic compounds from aqueous samples by separatory funnel liquid-liquid extraction.

2.0 SUMMARY OF METHOD

2.1. A measured volume of sample, usually 1 liter, at a specified pH is serially extracted with methylene chloride using a separatory funnel. The extract is dried, concentrated, and if necessary exchanged into a solvent compatible for extract cleanup or the determinative analysis method.

2.2. The routine extraction conditions for various determinative methods are given on extraction logs known as bench sheets that are created for each determinative method department supervisor. The bench sheet for each method includes the following information: routine extraction sample volume or weight, surrogate/spike name, concentration and volume, initial extraction pH, extraction solvent, exchange solvent, final volumes and cleanup methods required.

2.3. This procedure is based on Method 3510C, Revision 3, December 1996.

3.0 DEFINITIONS

3.1. Definitions are included in Appendix A.

4.0 INTERFERENCES

4.1. Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure must be of reagent grade or high purity in order to minimize interference.

5.0 SAFETY

5.1. Employees must trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Specific Safety Concerns & Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggle when it is performed.

5.3. Primary Materials Used

Table 1, Section 18.0 lists materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1. Separatory funnel - 2 Liter, with Teflon stopcock.

6.2. Glass Funnel - 75mm

6.3. Kuderna-Danish (K-D) Apparatus:

Concentrator tube – 10 mL, graduated (Kontes 570051-1025 or equivalent).

Evaporation flask – 500 mL (Kontes 570037-0500 or equivalent).

Snyder column - Three-ball macro (AMK KD-0018 or equivalent).

Snyder column - Two-ball micro (AMK KD-0013 or equivalent).

6.4. Solvents Vapor Recovery System (Kontes K-545000-1006 or K-547300- 0000, Ace Glass 6614-30, or equivalent).

6.5. Boiling chips, Solvent extracted ~Approximately 10/40 mesh (silicon carbide or equivalent).

6.6. Water bath - Heated, capable of temperature control ±5°C.

6.7. pH indicator paper or pH meter

6.8. Adjustable Pipette - 0.5 to 2.0mL.

6.9. Graduated cylinder - 1 Liter. (Class A).

6.10. 8 micron fiberglass wool

6.11. Vials and caps: 1.8, 4, 8, and 16mL with Teflon lined septa and screw caps

6.12. Separatory Funnel Shaker

6.13. Labconco RapidVap and Rapid Trap Solvent Recovery System with 600ml glassware.

6.14. Concentrator Tubes, graduated 10 mL

7.0 REAGENTS AND STANDARDS

7.1. Reagents

Reagent water

Sodium Hydroxide Solution (6N), NaOH: Dissolve 240 g NaOH in reagent water and dilute to 1 Liter.

Sulfuric Acid Solution (1:1 v/v), H_2SO_4: Slowly add 50mL of H_2SO_4 to 50mL of organic-free reagent water.

Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400^{o}C for 4 hours.

Methylene chloride, CH_2Cl_2- Pesticide quality or equivalent.

Hexane, C6H14- Pesticide quality or equivalent.

Acetone, (CH3)2CO- Pesticide quality or equivalent.

7.2. Standards

Stock standard are purchased as certified solutions from commercial vendors. Surrogate and spike standards are prepared in the laboratory by diluting mixed stock standard solutions in solvent. The concentration of the standard solution and volume used is recorded on the extraction log. Prepared standard solutions are stored in glass containers at 4°C. Unless otherwise specified, prepared standards are assigned an expiration date of 6 months from date of preparation or in accordance with the expiration date of the parent standard, whichever is sooner.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1. Samples should be collected in 1 L amber glass bottles fitted with Teflon-lined screw caps. If amber containers are not available, alternate measures should be taken to protect the samples from light sources. Immediately following collection, samples should be iced to 4°C (±2°C) and maintained at that temperature until the time of extraction.

8.2. The extraction holding time is 7 days from the time of collection.

8.3. Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 A Method Blank (MB) and Laboratory Control Sample (LCS) are prepared with each extraction batch. These samples show that the laboratory is in control, independent of the sample matrix.

9.2 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) are prepared with each extraction batch. Project specific MS/MSD are performed per client request. Sample Duplicates (SD) are performed per client request. These samples show the effect of the sample matrix on the accuracy and precision of the method.

9.3 A Surrogate spike is added to all field and QC samples before extraction to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix.

9.4 The criteria used to assess quality control samples are given in the analytical SOP for the determinative method.

10.0 CALIBRATION AND STANDARDIZATION

10.1. Check the calibration of the pH meter each day of use prior to use.

10.2 Check the calibration of the balance each day of use prior to use.

10.3 Check the calibration of the adjustable pipettes each day of use prior to use.

11.0 PROCEDURE

11.1. Equipment Set Up

Prepare glassware using the procedures described in laboratory SOP LP-OP-Glass. Label the glassware for each sample and QC item clearly and unambiguously during each step of the extraction procedure. Solvent will erase grease pens and “sharpie ink”, caution must be taken to ensure labels are not obliterated during the procedure.

Assemble a drying funnel for each field and QC sample: Place a plug of glass wook in to a 75mm glass funnel. Add a sufficient amount of purified granular sodium sulfate sufficient to fill the funnel ¾ full. Rinse the funnel with ~30mL of acetone and discard the solvent rinse.

Assemble a KD setup for each filed and QC sample: Attach a 10 mL concentrator tube to a 500 mL evaporation flask, then attach to a pre-assembled drying funnel.

11.2. Extraction

Measure 1 L of sample into a graduated cylinder then quantitatively transfer the sample to the separatory funnel. Alternatively, if samples were received in 1 L containers, mark the meniscus of the aqueous on the sample container (1L) with a permanent marker. Pour the entire sample into the 2 L separatory funnel. Rinse the sample container with ~60 mL methylene chloride and pour the rinsate into the extractor. To measure the actual sample volume, fill the sample container with tap mater to the mark of the meniscus and pour the water into a graduated cylinder for volume measurement. Record the sample volume on the extraction benchsheet. For the method blank and laboratory control sample, use 1L of reagent water.

Note: If high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to 1L with reagent water. If smaller volumes are used, it may be necessary to adjust the volume of surrogate and spike solution. Consult with the Department Supervisor for further guidance.

Add the proper type and volume of surrogate solution to each field and QC sample and add the proper type and volume of spike solution to the LCS and MS/MSD. (Refer to the extraction bench sheet for details).

Check the pH of each sample using a calibrated pH meter. Adjust the pH, if necessary to the pH using 1:1 sulfuric acid solution or 1:1 sodium hydroxide solution. Thoroughly rinse the probe of the pH meter with reagent water between each sample in order to avoid cross-contamination.

Cap and shake each separatory funnel vigorously for 15-20 seconds with periodic venting to release excess pressure. Place the separatory funnel on the mechanical wrist-action shaker and shake for 2 minutes.

Caution: Methylene chloride creates excessive pressure rapidly, initial venting must be performed immediately after the separatory funnel has been sealed and shaken once. Venting of the separatory funnel must be performed into a fume hood in order to avoid exposure to solvent vapors.

Allow sufficient time for the organic layer to separate from the water layer. If an emulsion forms and it is more than one-third the size of the solvent layer, filter or stir the extract to remove the emulsion.

Drain the solvent extract into the glass funnel / concentration apparatus and repeat the extraction two more times using fresh portions of methylene chloride. Combine the three extracts. If further pH adjustment and secondary extraction is required adjust the pH and repeat the extraction three more times.

Perform concentration using the Kuderna-Danish technique (KD)

11.3 K-D Technique

11.3.1. Macro Snyder Column

Add one or two clean boiling chips to the KD flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding ~10 mL of acetone to the top of the column. Place the K-D apparatus in a hot water bath at a temperature of 15-20° C above the boiling point of the solvent 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. Attach the solvent vapor recovery glassware to the Snyder column of the K-D apparatus following manufacturer's instructions. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches <10 mL, remove the K-D apparatus from the water batch and allow it to drain and cool for at least 10 minutes.

If a solvent change is required for cleanup or analysis, when the apparent volume of extract is approximately <10 mL, add ~60 mL of exchange solvent to the top of the Synder column. Continue to concentrate the extract to an apparent volume of less than <10mL. Remove the Synder column and rinse the flask and the lower joints into the concentrator tube with 1-2 mL of methylene chloride or the exchange solvent. Adjust the extract to final volume. If the final extract volume should be less than 10 mL, concentrate the extract further using a micro-Synder column.

***Caution**: Extracts designated for 8270 PAH analysis should not be allowed to concentrate to less than 1mL in order to minimize volatilization of target compounds. When concentrating EPH's, do not use nitrogen.*

11.3.2 Micro-Snyder Column

Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column.

Place the concentrator tube into the water bath so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the concentrator tube and/or the temperature of the water bath to ensure that concentration of the extract is complete within 5-10 minutes. Continuously monitor the distillation process to ensure sample extracts do not evaporate too quickly. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with solvent. When the apparent volume reaches 0.5-1.0 mL, remove the K-D apparatus from the hot water bath and allow it to drain and cool for at least 10 minutes. Remove the Synder column and rinse the flask and the lower joints into the concentrator tube with 0.2 mL of methylene chloride or the exchange solvent. Adjust to final extract volume.

11.3.3 Nitrogen Blow Down

Use nitrogen blowdown when concentration using the K-D technique may be difficult due to high extract viscosity. Place the concentrator tube in a warm bath (65°C) and apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to

prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

11.4 Extract Preparation & Handling

Transfer the extract into a labeled Teflon lined screw or crimp-top vial.

If extract cleanup is not required, complete the extraction bench sheet and enter the extraction data into the Organic Extraction Module in LIMS, attach the associated paperwork and submit with extracts to the department supervisor for review. If review will not be performed immediately, place the extracts in refrigerated storage. After review is complete, relinquish the extracts to the appropriate analytical department.

If extract cleanup is required, complete the extraction bench sheet and place the paperwork in the location designated for work scheduling of extract cleanup procedures. If cleanup will not be performed immediately, place the extracts in refrigerated storage.

12.0 CALCULATIONS

12.1 The equations used to derive analytical results are provided in the test method SOP for analysis.

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1. Complete the extraction bench. Document problems encountered during the extraction process are documented on the bench sheet or complete a nonconformance report, if necessary. Set aside the extracts for final project check by the department supervisor. For additional guidance regarding the laboratory's protocol and required elements for each level of data review refer to laboratory SOP LP-LB-003 *Data Review*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009 *Method Detection Limits.*

14.2 A demonstration of analyst capability (IDOC) is required before use of this SOP and any time there is a significant change in instrument type, personnel or test method.

14.3 Employee Training, and IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and

the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 Waste Streams Produced by the Method

The following waste stream is produced when this procedure is performed:

- Aqueous Waste-Satellite Container: 55 Gallon Steel or Poly Aqueous Waste Drum
- Solvent Waste -Satellite Container: 55 Gallon Steel Waste Drum

Waste streams produced in the laboratory are accumulated in satellite containers located in the work area. The satellite containers are labeled "Hazardous Waste" along with the type of waste category generated. Authorized personnel routinely transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in laboratory SOP LP-LB-001 *Hazardous Waste*.

16.0 REVISION HISTORY

16.1 Title Page: The Laboratory Directory was changed from Michael F. Wheeler to Christopher A. Ouellette. The Department Manager was changed from Jon K. Zygmuntowicz to Jennifer L. Clements.

16.2 Section 11.0: Procedures for rapid vap concentration was removed.

16.3 Section 16.0 was renamed revision history (formerly references).

16.4 Section 17.0 was renamed references (formerly tables, diagrams, flowcharts).

16.5 Section 18.0 was inserted as tables, diagrams, flowcharts.

17.0 REFERENCES

17.1. SW-846 Method 3510C, Revision 3, December 1996. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, Final Update III, December 1996.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

17.1. Table 1: *Primary Materials Used*
17.2 Appendix A: *Terms and Definitions*

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
[1]Always add acid to water to prevent violent reactions.			
[2]Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect, or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix: the substrate of a test sample.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a pure substance with properties that mimic the analyte of interest but that is unlikely to be found in environmental samples.

Test Method: defined technical procedure for performing a test.

SOP CHANGE -IN-PROGRESS ATTACHMENT (CIPA)

SOP Title: Ultrasonic Extraction
SOP No: LM-OP-3550
Revision: 3
Date Effective: 12/12/05
CIPA Date Effective: 02/27/06

QA Manager: Kirstin McCracken **Date:** February 27, 2006

Kirstin McCracken

Organic Manager: Jennifer L. Clements **Date:** February 27, 2006

Jennifer L. Clements

The following revisions or additions in BOLD TEXT were made to the referenced SOP. These changes were implemented on the CIPA Date Effective indicated above.

11.3.3 Nitrogen Blow Down

Use nitrogen blow down when concentration using the K-D technique may be difficult due to high extract viscosity. Place the concentrator tube in a warm bath (**~~65~~ 35**°C) and apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

STANDARD OPERATING PROCEDURE
ULTRASONIC EXTRACTION
SW-846 3550B
Applicable Matrix: Soil, Sludge, Waste & Tissue

APPROVAL SIGNATURES

Laboratory Director: _____ Date: December 12, 2005
Christopher A. Ouellette

QA Manager: _____ Date: December 12, 2005
Kirstin L. McCracken

Organics Manager: _____ Date: December 12, 2005
Jennifer L. Clements

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1.0 SCOPE AND APPLICATION

1.1. This SOP describes the laboratory procedure for isolating nonvolatile and semivolatile organic compounds from solids such as soils, sediments and waste by ultrasonic extraction. This SOP also describes the concentration technique used to prepare the extract for analysis by the appropriate determinative methods.

2.0 SUMMARY OF METHOD

2.1. Low Concentration

A measured weight of sample, usually 30 g, is mixed with anhydrous sodium sulfate to create a free-flowing mixture and solvent extracted three times using ultrasonic extraction. The extract is dried, concentrated, and if necessary exchanged into a solvent compatible for extract cleanup or the determinative analysis method.

2.2. Medium/High Concentration

A 2 g subsample is mixed with anhydrous sodium sulfate to create a free-flowing mixture and solvent extracted three times using ultrasonic extraction. The extract is dried, concentrated, and if necessary exchanged into a solvent compatible for extract cleanup or the determinative analysis method.

2.3. The routine extraction conditions for various determinative methods are given on document controlled extraction logs (also known as “benchsheets”) that are created for each determinative method by the Organic Extraction Department Manager or his designee. The benchsheet for each method includes the following information: routine extraction sample volume or weight, surrogate/spike name, concentration and volume, initial extraction pH, extraction solvent, exchange solvent, final volumes and cleanup methods required.

2.4. This procedure is based on Method 3550B, Revision 2, December 1996.

3.0 DEFINITIONS

3.1. Definitions are included in Appendix A.

4.0 INTERFERENCES

4.1. Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference. All glassware must be cleaned in accordance with laboratory SOP LP-OP-Glass and rinsed with acetone prior to use.

5.0 SAFETY

5.1. Employees must trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Specific Safety Concerns & Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

5.3. Primary Materials Used

Table 1, Section 18.0 lists materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1. Ultrasonic disrupter, Tekmar 600-watt Model Dual Output with pulsing capability, 3/4" standard disrupter horn for low level extractions and a 1/8" disrupter horn for medium level extractions.

6.2. Sonication box

6.3. Kuderna-Danish Apparatus:

Concentrator tube - 10mL, graduated (Kontes 570051-1025 or equivalent).

Evaporation flask - 500mL (Kontes 570037-0500 or equivalent)

Snyder column- Three Ball Macro (AMK KD-0011); Two Ball Micro (AMK KD-0013).

6.4. Disposable glass Pasteur pipettes and bulb.

6.5. Balance - capable of measuring 100g to ± 0.01g.

6.6. Filter Funnels - 100mm diameter for filtration/drying.

6.7. Filter paper - No. 541 Whatman 18.5cm, or equivalent.

6.8. Beakers - 400mL.

6.9. Stainless steel or Teflon spatulas.

6.10 Adjustable pipette

6.11. 5 and 10mL comparison vials.

6.12. Vials and caps: 1.8, 4, 8, and 16mL with Teflon lined septa and screw caps.

7.0 REAGENTS AND STANDARDS

7.1. Reagents

Reagent water.

Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400^{o}C for 4 hours.

Methylene chloride, CH_2Cl_2- Pesticide quality or equivalent.

Hexane, C6H14- Pesticide quality or equivalent.

Acetone, (CH3)2CO- Pesticide quality or equivalent.

1:1 Methylene Chloride/Acetone: Mix 2 L methylene chloride with 2 L acetone. Store in a 4L amber glass bottle.

1:1 Hexane /Acetone: Mix 2 L hexane with 2 L acetone. Store in a 4 L amber glass bottle.

7.2. Standards

Stock standard are purchased as certified solutions from commercial vendors. Surrogate and LCS/matrix spike standards are prepared in the laboratory by diluting mixed stock standard solutions in solvent. The concentration of the standard solution and volume used is recorded on the extraction log. Prepared standard solutions are stored in glass containers at 4°C. Unless otherwise specified, prepared standards are assigned an expiration date of 6 months from date of preparation or in accordance with the expiration date of the parent standard, whichever is sooner.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1. Samples should be collected in 250 mL wide mouth-amber glass jars. Immediately following collection, samples must be iced to 4^{o}C (±2) and maintained at that temperature until time of extraction.

8.2. The routine extraction holding time for most methods is 14 days from date of collection.

8.3. Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 A Method Blank (MB) and Laboratory Control Sample (LCS) are prepared with each extraction batch. These samples show that the laboratory is in control, independent of the sample matrix.

9.2 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) are prepared with each extraction batch. Project specific MS/MSD are performed per client request. Sample Duplicates (SD) are performed per client request. These samples show the effect of the sample matrix on the accuracy and precision of the method.

9.3 A Surrogate spike is added to all field and QC samples before extraction to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix.

9.4 The criteria used to assess quality control samples are given in the analytical SOP for the determinative method.

10.0 CALIBRATION AND STANDARDIZATION

10.1. Check the calibration of the pH meter each day of use prior to use.

10.2 Check the calibration of the balance each day of use prior to use.

10.3 Check the calibration of the adjustable pipettes each day of use prior to use.

10.4 Check the tune of the sonicator horn prior to use and inspect the horn tip for excessive wear. Attach the converter to the bottom connector. Set the pulsar switch to the off position, the output control to 10 (full power) and the % duty cycle to 50 (energy on 50% of time and off 50% of time). Briefly depress the tune switch and rotate the tuning control clockwise or counterclockwise until a minimum reading of less than 20% is achieved on the power meter. Re-attach the upper converter if using both converters and record the tune calibration in the logbook designated for this purpose.

11.0 PROCEDURE

11.1. Equipment Set Up

Assemble equipment and ensure that the glassware has been cleaned and pre-rinsed in accordance with laboratory SOP LM-OP-Glass. Label samples and QC items clearly and unambiguously during each step of the extraction procedure. Solvents will erase grease pens and “sharpie ink” caution must be taken to ensure that the labels are not obliterated during the procedure.

Assemble a K-D concentrator for each field and QC sample by attaching a 10mL concentrator tube to a KD flask.

Prepare a drying funnel for each sample. Rinse the funnel with Acetone, fold a 185mm Whatman® 54 filter into quarters and place the filter in the funnel. Add baked anyhydrous sodium sulfate to each funnel until the funnel is ~3/4 full. Rinse the funnel with approximately 30mL acetone and discard the solvent rinse. Place the prepared funnel onto each K-D setup.

Ensure the extraction horn is appropriate for the extraction protocol (See 6.1) because the extractions horns are not interchangeable between protocols.

11.2. Extraction

Mix each sample thoroughly and discard any foreign objects such as sticks, leaves, and rocks. Place a 400mL glass beaker onto the calibrated balance and tare the balance. Measure 30 g of sample (±0.05 g) into the beaker and record the weight on the extraction log. Blend the sample with a sufficient amount of baked anhydrous sodium sulfate to ensure a free-flowing mixture.

Note: Percent dry weight of solid samples is determined by the inorganic department following SOP LM-WC-Percent Solids.

Add the proper type and volume of surrogate solution to each field and QC sample and add the proper type and volume of spike solution to the LCS and MS/MSD. (Refer to the extraction bench sheet for details).

Add 100 mL of the extraction solvent or solvent mixture to each field and QC sample. For GC Methods (8081 & 8082), use 1:1 Acetone/Hexane extraction solvent. For GC/MS and DRO, use 1:1 Methylene Chloride/Acetone.

Clean the tip of the sonicator horn using a Kimwipe wetted with acetone. Place the bottom tip of the sonicator horn ~1/2 inch below the solvent, but above the sediment layer. Close the door to the sonicator box.

Set the control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy) and percent duty cycle knob set at 50% (energy on 50% of time and off 50% of time) extract ultrasonically for 3 minutes. Watch the extraction to ensure there is very active mixing of the sample and the solvent when the ultrasonic pulse occurs.

Decant the extract into the drying funnel. Repeat the extraction two more with two additional 100mL portions of extraction solvent. Decant the solvent between each extraction. After the final extraction, pour the entire sample into the funnel and rinse with ~30mL of extraction solvent. Allow the solvent to completely drain through the funnel into the K-D apparatus. Remove the funnel and discard the contents.

Concentrate the extract using K-D technique.

11.3. K-D Technique

11.3.1 Macro Snyder Column

Add one or two clean boiling chips to the KD flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding ~10 mL of acetone to the top of the column. Place the K-D apparatus in a hot water bath at a temperature of 15-20° C above the boiling point of the solvent 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. Attach the solvent vapor recovery glassware to the Snyder column of the K-D apparatus following manufacturer's instructions. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches <10 mL, remove the K-D apparatus from the water batch and allow it to drain and cool for at least 10 minutes.

If a solvent change is required for cleanup or analysis, when the apparent volume of extract is approximately <10 mL, add ~60 mL of exchange solvent to the top of the Synder column. Continue to concentrate the extract to an apparent volume of less than <10mL. Remove the Synder column and rinse the flask and the lower joints into the concentrator tube with 1-2 mL of methylene chloride or the exchange solvent. Adjust the extract to final volume. If the final extract volume should be less than 10 mL, concentrate the extract further using a micro-Synder column.

***Caution**: Extracts designated for 8270 PAH analysis should not be allowed to concentrate to less than 1mL in order to minimize volatilization of target compounds.*

11.3.2 Micro-Snyder Column

Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column.

Place the concentrator tube into the water bath so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the concentrator tube and/or the temperature of the water bath to ensure that concentration of the extract is complete within 5-10 minutes. Continuously monitor the distillation process to ensure sample extracts do not evaporate too quickly. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with solvent. When the apparent volume reaches 0.5-1.0 mL, remove the K-D apparatus from the hot water bath and allow it to drain and cool for at least 10 minutes. Remove the Synder column and rinse the flask and the lower joints into the concentrator tube with 0.2 mL of methylene chloride or the exchange solvent. Adjust to final extract volume.

11.3.3 Nitrogen Blow Down

Use nitrogen blowdown when concentration using the K-D technique may be difficult due to high extract viscosity. Place the concentrator tube in a warm bath (65°C) and apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

11.4 Extract Preparation & Handling

Transfer the extract into a labeled Teflon lined screw or crimp-top vial and store the extract refrigerated in preparation for cleanup or analysis.

If extract cleanup is not required, complete the extraction bench sheet and enter the extraction data into the Organic Extraction Module in LIMS, attach the associated paperwork and submit with extracts to the department supervisor for review. If review will not be performed immediately, place the extracts in refrigerated storage. After review is complete, relinquish the extracts to the appropriate analytical department.
If extract cleanup is required, complete the extraction bench sheet and place the paperwork in the location designated for work scheduling of extract cleanup procedures. If cleanup will not be performed immediately, place the extracts in refrigerated storage.

12.0 CALCULATIONS

12.1 The equations used to derive analytical results are provided in the test method SOP for analysis.

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1. Complete the extraction bench. Document problems encountered during the extraction process are documented on the bench sheet or complete a nonconformance report, if necessary. Set aside the extracts for final project check by the department supervisor. For additional guidance regarding the laboratory's protocol and required elements for each level of data review refer to laboratory SOP LP-LB-003 *Data Review*.

14.0 METHOD PERFORMANCE

14.1 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-LB-009 *Method Detection Limits.*

14.2 A demonstration of analyst capability (IDOC) is required before use of this SOP and any time there is a significant change in instrument type, personnel or test method.

14.3 Employee Training, and IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 Waste Streams Produced by the Method

The following waste stream is produced when this procedure is performed:

- Solid Waste – Satellite Container: 5 Gallon Polyethylene Bucket
- Solvent Waste- Satellite Container: Steel 55 Gallon Drum

Waste streams produced in the laboratory are accumulated in satellite containers located in the work area. The satellite containers are labeled "Hazardous Waste" along with the type of waste category generated. Authorized personnel routinely transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in laboratory SOP LP-LB-001 *Hazardous Waste*.

16.0 REVISION HISTORY

16.1 Title Page: The Laboratory Directory was changed from Michael F. Wheeler to Christopher A. Ouellette. The Department Manager was changed from Jon K. Zygmuntowicz to Jennifer L. Clements.

16.2 Section 16.0 was renamed revision history (formerly references).

16.3 Section 17.0 was renamed references (formerly tables, diagrams, flowcharts).

16.4 Section 18.0 was inserted as tables, diagrams, flowcharts.

17.0 REFERENCES

17.1. SW-846 Method 3550B, Revision 2, December 1996. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, Final Update III, December 1996.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

17.1. Table 1: *Primary Materials Used*
17.2. *Appendix A:* Terms & Definitions

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
[1]Always add acid to water to prevent violent reactions.			
[2]Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect, or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix: the substrate of a test sample.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a pure substance with properties that mimic the analyte of interest but that is unlikely to be found in environmental samples.

Test Method: defined technical procedure for performing a test.

STANDARD OPERATING PROCEDURE
GEL-PERMEATION CLEANUP
SW-846 3640

Applicable Matrices: Solid and Chemical Materials, Biological Tissue

Approval Signatures

Laboratory Director: Michael F. Wheeler, Ph.D. Date: 5/20/05

QA Manager: Kirstin L. McCracken Date: 5/20/05

Department Manager: Jon K. Zygmuntowicz Date: 5/20/05

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedures for gel-permeation cleanup (GPC) applied to organic extracts in order to eliminate sample interferences that may cause errors in quantitation including false positive/false negative results, rapid deterioration of capillary columns and instrument downtime.

1.2 GPC is a size exclusion cleanup procedure that uses organic solvents and hydrophobic gels in the separation of synthetic macromolecules. The packing gel is porous and is characterized by the range or uniformity (exclusion range) of that pore size. The exclusion range must be larger than the molecular size of the molecules to be separated. GPC cleanup is recommended for the elimination from the sample of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular-weight compounds and is appropriate for both polar and non-polar analytes. The cleanup procedure separates high molecular, high boiling material from sample analytes and is useful for removing materials that contaminate injection ports and column heads. The cleanup procedure should not be used to eliminate extraneous peaks in a chromatogram that interfere with analytes of interest.

2.0 SUMMARY OF METHOD

2.1 Prior to the GPC cleanup procedure, samples are solvent extracted following approved laboratory standard operating procedures. A GPC column is packed with the required amount of pre-swelled absorbent, and is flushed with solvent for an extended period. The column is calibrated and then loaded with the sample extract to be cleaned up. Following cleanup, the sample is concentrated to the appropriate volume specified in the determinative method in preparation for analysis.

2.2 This SOP is based on SW846 Method 3640A, CLP SOW OLM03.2 and OLM04.2/4.3.

3.0 DEFINITIONS

3.1 This section is not applicable to this SOP.

4.0 INTERFERENCES

4.1 Variation in temperature may cause retention times and dump and collect times to shift. To prevent this, the laboratory should be maintained at a consistent temperature.

4.2 Highly viscous extracts may overload the GPC and should be diluted prior to cleanup. Sample extracts with a viscosity greater than that of a 1:1 glycerol: water solution should be diluted and loaded into several loops. Similarly, extracts containing more than 400 mg of nonvolatile residue per 10 mL of extract should be diluted and loaded into several loops.

4.3 Particles greater than 1 micron may scratch the valve, which may result in a system leak and cross contamination of sample extracts in the sample loops. To avoid this problem, sample extracts are filtered through a 0.45-micron filter disk prior to GPC cleanup.

4.4 Particulates or glass wool in the GPC switching valves can cause damage that would require factory repair.

5.0 SAFETY

5.1 Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Safety Concerns or Requirements

Protective clothing such as a lab coat, safety glasses and latex gloves should be worn when performing this procedure.

5.3. Primary Materials Used

Table 1, Section 17.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 GPC Systems

GPC Autoprep Model 1000, GPC Autoprep 1002B, Autoinject 110 assembled with the following components: an HPLC pump, an auto sampler or a valving system with sample loops, and a fraction collector.

6.2 Chromatographic column, ABC Heavy Barrel Glass Column, 700 mm x 25 mm.

6.3 Bio Beads (SX-3), 200-400 mesh, J2 Scientific)

6.4 Ultraviolet detector 254 nanometers fixed.

6.5 Strip chart recorder, 3392A HP Integrator

6.6 0.45 um Whatman Autovial

6.7 Graduated Cylinder

6.8 Dell Dimension 4400 PC

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Methylene Chloride; J. T. Baker, Pesticide quality or equivalent

Acetone; J. T. Baker, Pesticide quality or equivalent

Hexane; J. T. Baker, Pesticide quality or equivalent

7.2 Standards

Stock standard calibration solutions are purchased from commercial vendors. The working GPC calibration standard solutions are prepared in volumetric flasks by diluting a volume of the stock standard in acetone in order to obtain the appropriate concentration. The concentration and components for each standard solution is given below:

GPC Calibration Solution

Compound	mg/mL
Corn Oil	25.0
bis (2-ethylhexyl-phthalate)	1.0
Methoxychlor	0.1
Perylene	0.02
Sulfur	0.08

GPC Calibration Verification Solution (Pesticide)

Compound	mg/L
gamma-BHC	0.1
Heptachlor	0.1
Aldrin	0.1
4,4'-DDT	0.2
Endrin	0.2
Dieldrin	0.2

GPC Calibration Verification Solution (PCB)

Compound	mg/L
Aroclor 1016	2
Aroclor 1260	2

The solutions are stored in an amber glass bottle with Teflon lined screw cap at 4°C (±

2°C) and protected from light. Because refrigeration may cause the corn oil to precipitate, allow the calibration solution to equilibrate to the room temperature prior to use. Replace the calibration standard solution every six months, or more frequently as needed.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 This section is not applicable to this SOP.

9.0 QUALITY CONTROL

9.1 For all sample extracts that are cleaned using this procedure, the associated quality control samples are also processed through this cleanup procedure in the same manner as sample extracts.

10.0 CALIBRATION AND STANDARDIZATION

10.1 The GPC calibration procedure is described in Section 11.0.

11.0 PROCEDURE

11.1 GPC Column Preparation

Weigh 70 g of Bio Beads (SX-3) and transfer them to a quart bottle with a Teflon-lined cap or a 500 mL separatory funnel with a large bore stopcock, and add approximately 300 mL of methylene chloride.

Swirl the container to ensure the wetting of all beads. Allow the beads to swell for a minimum of 2 hours. Maintain enough solvent to cover the beads sufficiently at all times.

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, but no closer than 5 cm (measured from the gel packing to the collar).

Raise the end of the outlet tube to keep the solvent in the GPC column, or close the column outlet stopcock. Place a small amount of solvent in the column to minimize the formation of air bubbles at the base of poured column packing.

Swirl the bead/solvent slurry to get a homogeneous mixture and, if the wetting was done in a quart bottle, quickly transfer it to a 500 mL separatory funnel with a large bore stopcock. Drain the excess methylene chloride directly into the waste beaker, and 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, open the stopcock (if attached), 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 methylene chloride to just rewet the gel.

Wipe any remaining beads and solvent from the inner walls of the top of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

CAUTION: Do not tighten the seal if beads are between the seal and the glass surface because this can damage the seal and cause leakage.

Compress the column as much as possible without applying excessive force. Loosen the seal and gradually pull out the plunger. Rinse and wipe off the plunger. Slurry any remaining beads and transfer them into the column. Repeat and reinsert the 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, and loosen and remove the plunger as described. Repeat this procedure until the plunger is inserted successfully.

Push the plunger until it meets the gel, then compress the column bed about four centimeters.

Connect the column inlet to the solvent reservoir (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 1/16" OD x O.010" ID x 2". Pump methylene chloride through the column at a rate of 5 mL/min for one hour.

After washing the column for at least one hour, 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.

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 6-10 psi backpressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

When the GPC column is not to be used for several days, connect the column outlet line to the column inlet to prevent 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 recalibrate after column drying has occurred to verify retention volumes have not changed.

11.2 GPC Calibration Procedure

Each GPC system must be calibrated when the calibration verification does not meet acceptance criteria, each time the GPC column is changed and once every seven days.

Establish the GPC operating conditions following the manufacturer's instructions. Prepare the calibration solution at the concentration specified in Section 7.2.

Establish the flow rate of the GPC by pumping solvent through the column for 5 minutes and collect the eluate in a 50 mL graduated cylinder.

Load the calibration solution onto the GPC following the manufacturers instructions. Evaluate the UV trace against the following technical acceptance criteria. If criteria are not met, investigate and correct the problem, if necessary prepare a new GPC column.

- Symmetrical peaks must be observed for all compounds in the calibration solution
- Corn oil and phthalate peaks must exhibit a >85% resolution
- Phthalate and Methoxychlor peaks must exhibit a >85% resolution
- Methoxychlor and Perylene peaks must exhibit a >85% resolution
- Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.
- The retention times for bis (2-ethylhexyl phthalate) and Perylene must not vary by more than ±5% between calibrations.

Using the information from the UV trace, determine the "collect" and "dump" times:

Pesticides/PCBs:

Establish a "dump" time that will remove >85% of the phthalate and a "collect" time that collects >95% of the Methoxychlor. Continue the collect until just prior to the elution of sulfur. Use a "wash" time of 15 minutes after the elution of Perylene.

Semivolatiles:

Initiate collection just before elution of bis (2-ethylhexyl) phthalate and after the elution of corn oil. Stop the collection shortly before the elution of sulfur. Use a "wash" time of 12 minutes after the elution of sulfur.

After the collect and dump cycles have been set, establish the flow rate and column pressure. Reinject the calibration solution and collect the GPC eluate in a graduated cylinder, stop the collect after 10 minutes and measure and record the volume. The volume of eluate should be within the manufacturer specified range of 4.5-5.5 mL/min. If the flow rate is outside this range, adjust the pump speed and repeat the procedure. Once the flow rate is within range, record the column pressure, which should be within the manufacturer specified range of 6-10 psi.

Analyze a GPC blank of methylene chloride. Concentrate the GPC blank using K-D

evaporator. For pesticide/PCB, exchange the solvent blank to hexane and concentrate to 5.0 mL. Relinquish the concentrated blank to the analytical department for analysis. The analyte concentrations in the GPC blank must be less than the reporting limit or CRQL (CLP SOW) for any target compound. If it is not, pump additional amounts of methylene chloride through the column and analyze another GPC blank to ensure the system is clean.

11.3 GPC Calibration Verification (CLP SOW)

Calibration verification must be performed once every 7 days immediately following the GPC calibration whenever samples designated for analysis by the CLP SOW (including QC) are to be cleaned up using the GPC.

NOTE: GPC calibration verification is not required for samples extracts designated for analysis by SW-846 methods.

Prepare the GPC calibration verification solutions following the procedure given in Section 7.2.

Pesticide/PCB:

Inject 10 mL of the pesticide GPC calibration verification solution into the first sample loop of the system. Load the Aroclor mixture onto the system in the same manner. Initiate the GPC program that was established during the calibration procedure. Concentrate the fractions using K-D technique, solvent exchange into hexane and adjust the final volume to 10 mL.

Relinquish the extract to the analytical department for analysis. The recovery of each of the single component analytes must be between 80-110 percent and the Aroclor patterns must be the same as those from the AR1016 and AR1260 standards in the initial calibration sequence. If the criteria are not met, the GPC column should be replaced and recalibrated before proceeding with cleanup of any samples.

11.4 Sample Cleanup

Wash all glassware following SOP LM-OP-Glass and rinse the GPC input tubes with acetone and methylene chloride.

The solvent extract must be primarily methylene chloride. All other solvents, e.g.1:1 methylene chloride/acetone, must be concentrated to 1 mL and diluted to 10 mL with methylene chloride prior to GPC Clean-up.

Prior to GPC cleanup, filter each extract through a 0.45 micron Autovial into a 16 mL amber vial with a Teflon lined cap.

Set up the GPC following the manufacturer instructions.

Prepare a collection bottle for each sample, and QC item. Label each bottle with the laboratory ID. Place the collection bottle into position and place a collection line into the collection bottle. Fill the input tubes with sample and place into the appropriate GPC injection position.

Process each sample using the collect and dump times established during GPC calibration. After the GPC fraction has been collected, concentrate the extract, solvent exchange pesticide/PCB samples into hexane and relinquish the extracts to the appropriate analytical department along with the associated paperwork or schedule and perform additional cleanup procedures when required.

12.0 CALCULATIONS

12.1 The GPC cleanup procedure processes only half of the sample extract loaded onto the GPC (5 mL of the 10 mL extract), the other half is "lost" with the solvent elution. Thus, a dilution factor of 2 must be used for the quantitation of the sample in the determinative method.

13.0 DATA ASSESSMENT, CRITERIA AND CORRECTIVE ACTION

13.1 Primary review is performed by the analyst(s) that performed the procedure. The data undergoes secondary review by a senior analyst, the department coordinator or a data review analyst. Problems encountered during analysis are documented and reported in the case narrative provided with the data package report.

14.0 METHOD PERFORMANCE

14.1 This section is not applicable to this procedure.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is carried out.

- Methylene Chloride Waste
- Cyclohexane waste

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

16.1 *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846),* Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

16.2 *Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration OLM04.2/4.3, OLM04.1, OLM03.2*_USEPA Contract Laboratory Program.

17.0 TABLES, DIAGRAMS, & FLOWCHARTS

17.1 Table 1: Primary Materials Used

Table 1: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

STANDARD OPERATING PROCEDURE
STL BURLINGTON

EXTRACT CLEANUP PROCEDURE
SW-846 Methods:

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedures for cleanup applied to organic extracts. Cleanup eliminates sample interferences that may cause errors in quantitation and identification including false positive/false negative results, rapid deterioration of capillary columns, and instrument downtime. The appropriate cleanup methods that should be used for a particular group of analytes are specified in the determinative methods. The following sections give a brief description and applicability of the cleanup methods included in this SOP.

Fractionation Cleanup and Adsorption Cleanup by Florisil and Silica Gel (Source: SW-846 3620B, 3630C, and 3600C)

Fractionation is a cleanup technique that can be used to separate complex mixtures of analytes such as PCBs from organochlorine pesticides (silica gel) and fractionation of the organochlorine pesticides (florisil). Adsorption chromatography is used to separate analytes of a narrow polarity range from interfering compounds of a different polarity. This technique is most commonly used to separate relatively non-polar compounds such as organochlorine pesticides, polynuclear aromatic hydrocarbons, and nitrosamines from more polar compounds. Florisil is a trade name for magnesium silicate (Mg_2SiO_3). Silica gel, also referred to as silicic acid, is made from sodium silicate and sulfuric acid.

Copper Cleanup (Source: SW-846 3660B and 3600C)
Elemental sulfur is extracted from many sediment samples, industrial waste, some soil samples, and even aqueous samples. Sulfur is extracted along with organochlorine and organophosphorous pesticides and PCBs and sulfur interference can interfere with or completely mask target analyte peaks in the early region of the chromatogram. Organic extracts are treated with activated copper granules or powder to remove sulfur prior to analysis.

Sulfuric Acid (Source: SW-846 3665A and 3600C)
Sulfuric acid is an effective cleanup of sample extracts to be analyzed for polychlorinated biphenyls (PCBs) and should be used whenever elevated baselines or overly complex chromatograms would prevent accurate quantitation or identification of PCBs. This procedure cannot be used to cleanup extracts to be used for analysis of other target analytes as it will destroy most organic chemicals including the pesticides Aldrin, Dieldrin, Endrin, Endosulfan I and II, and Endosulfan Sulfate. It is important to note that the extract must be solvent exchanged to hexane before adding concentrated sulfuric acid as a reaction with other solvents can be violent and dangerous.

2.0 SUMMARY OF METHOD

2.1 Prior to the cleanup procedure, samples are solvent extracted following approved laboratory standard operating procedures. Following cleanup, the sample is concentrated to the appropriate volume specified in the determinative method in preparation for analysis.

2.2 This SOP is based on the following reference methods:

SW-846 3600C (Cleanup)
SW-846 3620B (Florisil)
SW-846 3630C (Silica Gel)
SW-846 3660B (Sulfur)
SW-846 3665A (Sulfuric Acid)

3.0 DEFINITIONS

3.1 This section is not applicable to this SOP.

4.0 INTERFERENCES

4.1 Florisil Cleanup

The method developer found that phthalate ester contamination was detected at levels up to 400ng/cartridge when this cleanup procedure was evaluated; complete removal may not be possible.

4.2 Silica Gel Cleanup

Phthalate ester contamination may be present when using silica gel cartridges. Use of and inert column or cartridge (made of glass or polytetrafluoroethylene (PTFE)) may reduce this contamination.

4.3 Copper Cleanup

There are no interferences that are intrinsic to the copper cleanup procedure, but an instrument blank or the preparation blank should be subjected to the cleanup procedure to ensure that the process did not introduce contamination to the extracts. Additionally, if activated copper powder is used, the acid used to activate the copper must be completely removed as the acid could degrade some target analytes.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 Specific Safety Concerns or Requirements

5.3 Primary Materials Used

Table 1, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. This table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0.

Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 Florisil Cleanup

- Vacuum Manifold, Supelco 24 port.
- Valve Liners- Supelco
- Vacuum Pump

6.2 Silica Gel Cleanup

- 3 g column (10.5 ID, 13 OD 250 mm length)
- 10 g column (14.5 ID, 17 OD, 250 mm length)
- Glass Wool
- Glass Rod(s)
- 50 mL Beakers

6.3 Copper Cleanup (Copper Powder Activation)

- 8 mL/16 mL/40 mL Glass Vials with Teflon lined Caps
- Vortex Mixer
- Spatulas
- Disposable glass pipettes
- Centrifuge

6.4 Sulfuric Acid Cleanup

- Glass Vials with Teflon lined Caps
- Vortex Mixer
- Centrifuge, HN-S
- Brinkmann Dispenser
- Disposable glass pipettes

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Florisil

Acetone; J.T. Baker, Pesticide quality or equivalent.

Hexane; J.T. Baker, Pesticide quality or equivalent.

Florisil Cartridge: 6 mL/1gm Florisil Cartridge, Restek # 24034 or equivalent.

Hexane/Acetone (90:10 v/v): Measure 450 mL of hexane and 50 mL of acetone into a 500 mL labeled amber jar. Prepare the reagent fresh each day the cleanup method is performed.

7.1.2 Silica Gel

Silica Gel, 100-200 Mesh, Fisher # S679-500 or equivalent.

Reagent Water

3% Deactivated Silica Gel: Bake silica gel in an oven set at a temperature of 130°C for 8 hours. Weigh a 500 mL amber jar, add baked silica gel and weigh. Cap the jar and allow silica gel to cool. Add reagent water equal to 3% of the weight of the baked silica gel and tumble for 4 hours. Store tightly sealed in amber jar. Set expiration at 6 months from the date tumbled.

Sodium Sulfate Anhydrous, J.T.Baker Granular Cat# 3375-09

Methylene Chloride; J.T. Baker, Pesticide quality or equivalent.

Hexane; J.T. Baker, Pesticide quality or equivalent.

Reagent Water

7.1.3 Copper Cleanup

Pre-Activated Copper Granules, Restek or equivalent. Alternatively, use in-house prepared activated copper powder.

Materials Needed for Activated Copper Powder:

- Copper Powder, Spheroidal, <10 microns, 99%, Aldrich or equivalent. To activate the copper powder:
- Sulfur Sublimed Powder
- Hydrochloric Acid Concentrated, J.T. Baker Cat# 9530-33.
- Methanol, Ultra- Resi Analyzed, J. T.Baker, Cat# 9263-03
- Methylene Chloride, J.T. Baker, Pesticide Quality or Equivalent
- Hexane, J.T. Baker, Pesticide Quality or Equivalent
- Reagent Water
- Nitrogen Gas

Activation Procedure:

1) Transfer 5 to 8 g of copper powder into a clear glass 40 mL vial.

2) Add 15-20 mL of HCl to the vial. Cap and vortex the vial for 1 minute.

3) Centrifuge the vial until the HCl is separated from the copper powder. Decant and discard the HCl.

4) Repeat Steps 2-3 with another portion of HCl, then twice with reagent water followed by methanol, methylene chloride and hexane.

5) Dry the treated copper powder with nitrogen. Add approximately 0.25 g of the treated copper powder to an 8mL vial that contains a saturated sulfur solution (0.5 g sulfur/10 mL Hexane). If the copper turns black, the copper is considered activated. If the copper stays red, discard and start over with a new portion of copper powder. Care should be taken to avoid contaminating items and surfaces with sulfur powder.

7.1.4 Sulfuric Acid

Hexane; J.T. Baker, Pesticide quality or equivalent

Sulfuric Acid-concentrated. J.T.Baker

7.2 Standards

Stock standard calibration solutions are purchased from commercial vendors. Working standards are prepared from stock standards by diluting a volume of stock standards in an appropriate solvent in volumetric flasks to obtain the appropriate concentration.

7.2.1 Florisil (Method 3620B)

2,4,5-Trichlorophenol Stock Standard (100 ug/mL), Ultra Scientific Cat# PH-260 or equivalent.

2,4,5-Trichlorophenol (0.1 ug/mL): Measure 0.5 mL of the 2,4,5-Trichlorophenol stock standard solution into a 500 mL volumetric flask that contains ~ 300 mL of acetone and adjust to volume with acetone. Transfer the solution to a labeled 500 mL amber jar. Assign an expiration date of 7 days and store at 4^{o}C.

Individual Standard Mixture A (INDA): Obtain from GC laboratory.

Florisil Cartridge Performance Check Mixture: Add 0.5 mL of the 0.1 ug/mL 2,4,5-Trichlorophenol solution and 0.5 mL of INDA to 4 mL of hexane. Reduce the mixture to a final volume of 0.5 mL using nitrogen.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 This section is not applicable to this SOP.

9.0 QUALITY CONTROL

9.1 Associated quality control samples must be processed through the cleanup procedure.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Check the accuracy of the adjustable pipettes on the day of use, prior to use.

11.0 PROCEDURE

11.1 Florisil Cleanup (SW-846 Method 3620)

11.1.1 Florisil Cartridge Performance Check

Check each lot of Florisil cartridges prior to use and every 6 months with the performance check solution.

1) Place a Florisil cartridge into a valve liner on the manifold. Condition the cartridge using the procedure given in 11.1.2. Place a KD concentrator tube underneath the frit and transfer 0.5 mL of the performance check mixture to the top of a conditioned cartridge. Elute with 9 mL of hexane/acetone (9:1 v/v). Rinse the cartridge 2 more times with 1 mL of the hexane/acetone mixture.

2) Remove the concentrator tube and reduce extract to a final volume of 1.0 mL using micro-synder KD concentration or nitrogen blow-down. Forward the performance check solution and an aliquot of the 2,4,5-Trichlorophenol spike solution to the GC department.

The lot of cartridges is acceptable for use if the if the percent recovery of 2,4,5-Trichlorophenol is less than 5%, the percent recovery of all pesticides is between 80-120%, and if there are no interfering peaks present. If criteria are not met, repeat the performance check.

11.1.2 Sample Cleanup by Florisil Cartridge

Place a valve liner insert into the vacuum manifold. Tighten the valve nut to ensure the valve liners are crimped shut. Attach a Florisil cartridge to the valve liner insert for each extract. Turn on the vacuum pump and set the vacuum to 10 in (254 mm) of Hg. Gravity flow may also be used. To condition the Florisil cartridge, open the valve nut and elute ~6 mL of to acetone through each cartridge then elute ~6 mL of hexane/acetone (9:1) through each cartridge. Close the valve nut to stop flow of solvent through. Do not allow cartridges to become dry at any point during the process. If cartridges go dry, repeat the conditioning step.

Release the vacuum and place a labeled 10 mL KD concentrator tube under each frit

inside the manifold. Check to ensure the solvent line from each cartridge is placed inside the appropriate concentrator tube as the top of the manifold is replaced.

If using vacuum flow, turn on the vacuum otherwise use gravity flow. Transfer 1 mL of extract to the top of a Florisil cartridge. Elute the extract through the cartridge using 9 mL of hexane/acetone (9:1) collecting the eluent in the concentrator tube. Do not let the cartridge go dry. Concentrate the extract using nitrogen blow down or micro-synder concentration technique to the final volume listed on the bench sheet. Transfer the extract to a labeled vial. Perform additional cleanup procedures as needed or relinquish the extract to the GC department.

11.2 Silica Gel Cleanup (SW-846 Method 3630C)

11.2.1 Column Preparation

Clean the silica column prior to use following the procedure in laboratory SOP LP-OP-GLASS. Add a plug of glass wool to the column. Close the stopcock and add sufficient methylene chloride to fill the column half-full. Tap the column or use a clean glass stir rod to remove any air bubbles. Add ~4 g of granular sodium sulfate to the column and tap the column to remove air bubbles.

Weigh an appropriate amount of 3% de-activated silica gel into a 50 mL beaker and add a sufficient amount of methylene chloride to form a slurry. The amount of deactivated silica gel depends on the determinative test method. In general, use 3 grams for PCBs and 10 grams for all other methods. Refer to the bench sheet to determine the appropriate amount. Stir the slurry with a glass pipette to remove air bubbles then quantitatively transfer the slurry to the column.

Open the stopcock to let the methylene chloride drain through the column. When the level of methylene chloride is just above the level of the silica gel, add approximately 4 g of sodium sulfate to the column. Drain the methylene chloride to the top of the sodium sulfate but **do not let the column go dry.** Add 60 mL of hexane to the column allowing the solvent to drain to approximately ½ inch from the top of the sodium sulfate.

11.2.2 Extract Cleanup

Transfer the full volume of extract volume to the top of silica gel column and elute through the column. **Do not let the column go dry.** Elute the column with the appropriate solvent and collect the solvent rinse in a KD concentrator set-up using the following protocol:

- For PAHs, rinse the column with 25 mL hexane, discard the rinsate. In succession, elute 25 mL of 10% of methylene chloride/hexane then elute 60 mL of 40% methylene chloride/hexane collecting the eluate in a KD concentrator tube. Concentrate the solvent using the KD technique to the appropriate final volume.

- For PCBs, add 60 mL of hexane to the column and collect the eluate in a KD concentrator tube. Concentrate the solvent using the KD technique to the

appropriate final volume.

- For other extractions methods please refer to the extraction benchsheet for the particular procedure to be used.

11.3 Copper Cleanup (SW-846 Method 3660)

Transfer the extract to a labeled 8 mL vial. Add approximately 0.25 g of activated copper powder or granuales to each vial using a stainless steel spatula. Cap the vial and vortex for approximately 1 minute. If sulfur is present, the copper will turn blackish in color. Repeat the procedure until the activated copper does not change in color. See benchsheet for final concentration and vialing instructions.

11.4 Sulfuric Acid (SW-846 Method 3665)

Transfer the extract to a 16 mL clear glass vial. Working in a fume hood, carefully add 5 mL of concentrated sulfuric acid to the vial. Check to ensure there is no gas or heat produced. If no reaction is observed, vortex the vial for one minute. The vortex of the extract/acid mixture must be visible in the glass vial to ensure that the fluids have mixed properly. Centrifuge the vial for at least one minute then examine the solvent layer. If the solvent layer is cloudy or highly emulsified, centrifuge for a longer period of time. If repeat mixing this does not separate the layers, cool the extracts then repeat centrifugation.

If the hexane layer is still colored, remove the acid layer (lower layer) and add a fresh 5 mL portion of sulfuric acid. Repeat the previous step until the acid layer is clear. Transfer to a concentrator tube, rinse the acid interface with 1 mL of hexane, add to initial volume removed from vial used for acid cleanup, then concentrate the extract to the final volume using nitrogen blow-down or micro-snyder concentration.

Note: Acid can destroy the GC column thus, it is important not to remove any acid with the hexane phase.

12.0 CALCULATIONS

12.1 This section is not applicable to this SOP.

13.0 DATA ASSESSMENT, CRITERIA AND CORRECTIVE ACTION

13.1 Primary Review: Review the bench sheet for correctness and completeness. Record any problems encountered during the extraction process on the bench sheet or complete a nonconformance report, when necessary. Set aside the extracts and paperwork for secondary review.

13.2 Secondary Review: Review the bench sheet against the preparation worksheet to ensure the procedure performed is consistent with project specifications. Authorize release of the extracts to the appropriate analytical department.

13.3 For additional guidance regarding the laboratory's protocol and required elements for data review refer to laboratory SOP LP-QA -019 *Data Review*.

14.0 METHOD PERFORMANCE

14.1 This section is not applicable to this SOP.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is performed:

- Hexane Waste-Satellite Container: 50 Gallon Waste Drum
- Acetone Waste-Satellite Container: 50 Gallon Waste Drum
- Mixed Solvent Waste-Satellite Container: 50 Gallon Waste Drum
- Methylene Chloride-Waste-Satellite Container: 50 Gallon Waste Drum
- Sulfuric Acid Waste-Satellite Container: 2.5L Waste Bottle Labeled with appropriate acid type (sulfuric).
- Solid Waste-Satellite Container: Solid Waste 10 Gallon Plastic Bucket (inside fume hood)

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in laboratory SOP LP-LB-001 *Hazardous Waste*.

16.0 REVISION HISTORY

The following changes were made in this revision:

16.1 Section 1.0: Changed references for background information about cleanup procedures and added language referencing the use of copper granules for cleanup.

16.2 Section 4.0: Changed interference information to only that concerning use of the procedures in the laboratory.

16.3 Section 6.0: Added language referencing the use of copper granules for cleanup.

16.4 Section 7.0: Added language referencing the use of copper granules for cleanup.

16.5 Section 11.0: Separated the procedures used for florisil check from florisil cleanup on

sample extracts and added the use of gravity as an alternative to vacuum for this procedure. Added procedure for the use of copper granules for cleanup.

16.6 Section 15.0: Added Acetone, Mixed solvent, and Solid Waste as addition waste streams. Removed Elemental Mercury Waste and Potassium Permananate Waste from the list of waste streams.

16.7 Section 16.0: Changed from “References” to “SOP Revision History”. Subsequent sections changed in turn.

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

18.0 TABLES, DIAGRAMS, & FLOWCHARTS

18.1 Table 1: Primary Materials Used, Hazards & Exposure Limits

Table 1: Primary Materials Used, Hazards, & Exposure Limits

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

ARCADIS BBL

Attachment 1-3

STL SW-846 Inorganic SOPs

STL BURLINGTON
STANDARD OPERATING PROCEDURE

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY
EPA Method 6010B
Applicable Matrix: Water, Soil, Sediment, and Sludge

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure used to determine trace elements and metals in solution derived from groundwater, TCLP and EP extracts, industrial and organic wastes, sediments and other solid waste samples that have been acid digested following the procedures given in the laboratory SOPs LM-MP-3050, LM-MP-3005, or LM-MP-3010. This SOP is applicable to the determination of total recoverable metals, dissolved metals and total metals.

1.2 The elements for which this procedure is applicable are given in Table 1 along with the routine reporting limit (RL). Elements and matrices other than those listed in Table 1 may be analyzed by this procedure upon client request if performance at the concentration levels of interest is demonstrated.

2.0 SUMMARY OF METHOD

2.1 Samples are acid digested using the appropriate procedure given in the laboratory SOP LM-MP-3050, LM-MP-3005, or LM-MP-3010. The digested samples are introduced to the ICP-AES, which measures characteristic emission spectra by optical spectrometry. An aliquot of sample is nebulized and the resulting aerosol is transported to a plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a spectrometric grating and the intensities of the emission lines are monitored by photosensitive devices. Background correction is performed with the background measured adjacent to analyte lines on samples during analysis. The sample is analyzed by multiple integrations (2) and the average integration is converted to a concentration from a calibration curve.

2.2 The procedure is based on SW846 Method 6010B, Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 2, December 1996.

3.0 DEFINITIONS

3.1 Total Recoverable Metals: The concentration of metals in an unfiltered sample following treatment with hot dilute mineral acid (Method 3005).

3.2 Dissolved Metals: The concentration of metals determined in a sample after the sample is filtered through a 0.45 µm filter (Method 3005).

3.3 Total Metals: The concentration of metals determined in a sample following digestion by Methods 3010 or 3050.

3.4 Definitions for general laboratory terms are included in Appendix B.

4.0 INTERFERENCES

4.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra. These effects are compensated by using computer correction of the raw data by monitoring and measurement of the interfering element and/or background correction adjacent to the analyte line.

4.2 Physical interferences are effects associated with sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies especially in samples that contain high dissolved solids and/or acid concentrations. The use of a peristalitic pump or sample dilution should minimize these interferences.

4.3 Chemical interferences such as molecular compound formation, ionization effects and solute vaporization effects are highly dependent on matrix type and specific analyte elements. These interferences are not typical with ICP-AES analysis but if observed, can be minimized by matrix matching, buffering the sample and careful selection of instrument operating conditions.

4.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample.

5.0 SAFETY

5.1 Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2 Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision, avoid looking directly at the plasma.

5.3 Primary Materials Used

Table 2, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 Inductively Coupled Argon Plasma Atomic Emission Spectrometer (ICP-AES); Thermo Jarrell-Ash Trace ICP-AES 61E or equivalent.

6.2 Volumetric Pipettes: Size 0.10-1.00 mL & 1.00-5.00 mL, Finpipette or equivalent.

6.3 Volumetric Flasks, Class A: Size 50, 100, 500, and 1000 mL.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Hydrochloric Acid (HCl), Concentrated: Reagent Grade, J.T. Baker or equivalent

Nitric Acid (HNO_3), Concentrated: Reagent Grade, J.T. Baker or equivalent

7.2 Standards

Stock standard solutions are purchased from commercial vendors and stored according to the manufacturer's recommendation. Intermediate and working standard solutions are prepared as needed and unless otherwise noted, they are assigned an expiration date of 6 months from date of preparation unless the parent standard expires sooner, in which case, the earliest expiration date is used. The recommended formulations for standards used in this procedure are provided in Appendix A.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT & STORAGE

8.1 Samples may be collected in either glass or plastic containers. The sample volumes required depend on the digestion procedure but the laboratory recommends that a minimum sample volume of 500 mL be used for water samples and for soils, 5 grams. Immediately following collection, water samples must be preserved with nitric acid to a pH less than 2. If dissolved metals are to be determined, the water sample should be filtered (on-site) prior to preservation.

8.2 The holding time is 180 days from date of collection.

8.3 Unless otherwise specified by client or regulatory program, after analysis, samples are held for 30 days and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 QC Requirements

The following QC samples are analyzed with each batch: Method Blank (MB), Laboratory Control Sample (LCS), Matrix Spike (MS), Sample Duplicate (SD), and a

Serial Dilution (5X). Sample results that exceed the linear range are diluted and reanalyzed.

In addition to analysis of the calibration standard and blank with every analytical sequence, instrument standardization is checked during the analytical run with a Second Source Standard (ICV), Low Level Standard (CRI), Continuing Calibration Verification (CCV), Calibration Blank (CCB) and Interference Check Solutions (ICSA, ICSAB).

For DoD samples, a Linear Range Varification (LRV) must be present somewhere in the analytical sequence. The LRV concentration must exceed that of the samples to be reported from the analysis. If sample results are below the concentration of the respective calibration standard, then the calibration standard level may be used as the linear range without actual determinative analysis.

The minimum frequency requirements, acceptance criteria and recommended corrective action for QC samples are summarized in Table 3, Section 18.0.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Operating Conditions

Set up the instrument with the proper operating conditions using the instructions provided by the instrument manufacturer. Operating manuals for each ICP-AES instrument are located in the laboratory and specific wavelengths are listed in Table 1, Section 18.0.

Perform plasma optimization per the manufacturer's instructions when a new instrument is set up or when there is a significant change in operating conditions in order to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array.

Establish and verify the interelement spectral interference correction routine (IECs) used during sample analysis. Verify the routine annually. To determine the IEC, analyze a single element standard for each element at 3 successive concentrations. For each element, document the presence of a positive or negative value of any other element whose absolute value exceeds the RL (interfering element). Calculate a "K" factor for each element by dividing the concentration found by the concentration of the interfering element. Take the average of the three "K" values for each interfering element and enter this value into the software system.

Determine the sensitivity (MDL), instrument detection limits (IDL), linear dynamic range and interference effects for each individual analyte line. Refer to laboratory SOP LP-QA-005 for additional guidance on the procedures for MDL and IDL studies. Determine MDLs annually and IDLs every 3 months.

Establish the upper limit of the linear dynamic range (LDR) for each wavelength used by determining the signal responses from a minimum of 2 different concentration standards

across the range. One of the standards should be near the upper limit. The %R should be within ± 10% of the known value. Establish new dynamic ranges when there is a significant change in instrument response and check the range every 3 months. *Note: for DoD work, samples must be either diluted to below the calibration standard level for each element of interest, or an LRV must be present somewhere in the analytical sequence. LRVs determined on a quarterly basis cannot be used.*

Profile the instrument by aspirating a 5 ppm solution of As and running the automatic profile routine. Adjust the spectrum shifter dial on the front of the instrument and re-run the profile routine until the peak position for As is within ±0.05 units from zero. Perform instrument maintenance when the intensity counts of the profile solution suddenly drop more than 2000 counts or when the total intensity drops by 5000.

10.2 Instrument Calibration

Calibrate the instrument daily according to the manufacturer's instructions with a calibration blank and the mixed-element calibration standard(s) following the procedure that begins in Section 11.1. Immediately following analysis of the calibration standards, analyze a Second Source Standard (ICV), Calibration Blank (ICB), Interference Check Solutions (ICSA, ICSAB), and the low level standard (CRI) solution. Analyze the continuing calibration verification (CCV) standard and blank (CCB). Repeat a CCV and CCB after every tenth sample and at the end of the sequence. The criteria for the instrument check standards are provided in Table 3, Section 18.0 along with recommended corrective actions.

10.3 Internal Standard Evaluation

Yttrium is used as an internal standard to compensate for matrix interferences. Check the response of the internal standard (IS) in every field and QC sample. The raw average should not vary by ± 30% from the raw average result of the ICB. If the IS response is outside this range, dilute and reanalyze the sample.

10.4 Troubleshooting: The following items can be checked in case of calibration failures:

Check the profile intensity with the analysis of a 5 ppm As profile solution. If the intensity has dropped by more than 2000 counts, remove and clean the torch. If the torch shows extreme signs of wear such as cracks, replace it. Change the peristaltic pump tubing and replace the transition tubing pieces (for example, those that connect the nebulizer to the mixing coil, if the tubing appears cloudy or discolored). Recheck the intensity of the profile solution to determine if maintenance performed was sufficient to correct the problem.

11.0 PROCEDURE

11.1 Standard & Sample Preparation

Transfer ~25 mL of each calibration standard [CAL #7, 4, 8, ICV, CCV, ICSA, ICSAB,

CRI] into individual, labeled autosampler tubes. Use 25 mL of mixed acid solution (5%HCl / 2%HNO3) for each calibration blank.

Transfer approximately 8 mL of each digestate to individual autosampler tubes. Prepare a serial dilution and post digestion spike using an aliquot of the un-spiked sample that was used for the matrix spike. To prepare the serial dilution, transfer 1.4 mL of parent sample to an autosampler tube and add 5.6 mL of mixed acid solution (5%HCl / 2%HNO3). To prepare the post digestion spike, transfer 0.08 mL of the matrix spike solution and 7.92 mL of parent sample to an autosampler tube.

11.2 Analysis

Allow the instrument to become thermally stable prior to analysis. Create a new autosampler template on the instrument PC and enter the sample IDs in the order of analysis. Place the samples, serial dilution, post-digestion spike, calibration blanks, mixed calibration standards, and performance check standards in the position on the autosampler rack that corresponds to their assigned position in the autosampler template. Place the autosampler rack in the autosampler tray and initiate the software macro to begin analysis.

An example analytical sequence is given below:

Calibration Blank
Calibration Standard #7
Calibration Standard #8
Calibration Standard #4
ICV
ICB
ICSA
ICSAB
CRI
LRV (DoD only)
CCV
CCB
10 Samples*
CCV
CCB
10 Samples*
CCV
CCB

**The number of samples between each CCB/CCV (10) includes the method blank, laboratory control sample, matrix spike, sample duplicate, serial dilution and the post digestion spike.*

After analysis is complete, review the results against the criteria given in Table 3, Section 18.0. Perform corrective action as needed and dilute and reanalyze any sample whose result exceeds the linear calibration range.

The ICP-AES software is configured to acquire a minimum of two replicate exposures for all analyses and to use the average result of multiple exposures for standardization. The data processing software calculates results and adjusts for appropriate factors such as dilution and dry weight. Equations used are given in Section 12.0.

12.0 CALCULATIONS

12.1 Water Sample Concentration

$$C_{(ug/L)} = \frac{\mu g}{L_{dig}} \times \frac{V_{dig}}{V_{samp}}$$

Where:
$\mu g/L_{dig}$ ICP result including dilution factors
V_{dig} Digestate Volume (mL)
V_{samp} Sample Volume (mL)

12.2 Soil/Sediment Sample Concentration

$$C_{(mg/Kg)} = \frac{\mu g}{L_{dig}} \times \frac{V_{dig}}{g_{samp}} \times \frac{100}{\%\ solids}$$

Where:
$\mu g/L_{dig}$ = ICP result including all dilution factors
V_{dig} = final digestate volume in Liters
g_{samp} = sample weight in grams

12.3 Percent Recovery (%R) LCS and CCVs

$$\%R = \frac{SR}{SA} \times 100\%$$

Where:
SR= Sample Result
SA=Concentration of Spike Added

12.4 Percent Recovery (%R) MS

$$\%R = \frac{SSR - SR}{SA} \times 100\%$$

Where:
SSR=Matrix Spike Result
SR=Sample Result
SA=Concentration of Spike Added

12.5 Relative Percent Difference (RPD)

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

Where:
D_1 = Sample result
D_2 = Duplicate Result

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Review the samples, standards and QC samples against the acceptance criteria given in Table 3, Section 18.0. If the results do not fall within the established limits or criteria, perform corrective action. If corrective action is not taken or unsuccessful, record the situation and flag the data. Primary review of the data is performed by the analyst(s) that performed the procedure. Secondary review is performed by a senior analyst or a data review analyst. All data that does not meet established criteria must be flagged with the appropriate data qualifier and noted in the project narrative.

14.0 METHOD PERFORMANCE

14.1 All analysts must perform an Initial Demonstration of Capability (IDOC) before unsupervised use of this procedure to analyze client samples. The DOC procedure is specified in the laboratory SOP for employee training.

14.2 Method Detection Limit (MDL) Study is required during initial method set-up and subsequently once per 12 month period following the procedures given in the laboratory SOP for the determination of MDLs. Instrument Detection Limits (IDL) should be determined every 3 months following the procedure given in laboratory SOP for the determination of IDLs. To comply with the requirement specified in the Department of Defense (DoD) Quality System Manual, IDL values must be less than or equal to the established MDL.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 Waste Streams generated by this method:

- Acid Waste

→ *Satellite Waste Container: 55 Gallon Closed Topped Poly labeled "CORROSIVE"*

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP for hazardous waste disposal.

16.0 REVISION HISTORY

16.1 Title Page: Changed to reflect current management team.
16.2 Section 9.1: Removed reference to Arizona requirements.
16.3 Section 10.1: Changed LRV limit from 5% to 10%. Added DoD LRV criteria. Added profile count criteria.
16.4 Section 10.2: Changed standard sequence to match that of example analytical sequence
16.5 Section 10.3 and 10.4: These sections were added.
16.6 Section 14.2: Added all except the first two lines of section 14.2 (from CIPA 01.00.06).
16.7 Section 18.0, Table 1: changed RLs for Fe, Pb, Se, Tl.
16.8 Section 18.0, Table 3: The frequency for the MS/MSD/SD was updated to reflect current practice. DoD requirements were changed to reflect current version of DoD QSM (from CIPA 01.00.06). Reference to Arizona work was removed. Removed MSD and added DoD LRV.
16.9 Section 18.0, Table 4: Control Limits for the CRI were added. changed from being CRI control limits required for Arizona work to DoD LRV concentrations.
16.10 Section 18.0, Appendix A: added new CRI intermediate and working standard tables to reflect current laboratory practice.

17.0 REFERENCES

17.1 SW846 Method 6010B, Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 2, December 1996.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Target Analyte List, RL and Wavelength Used by Instrument
18.2 Table 2: Primary Materials Used, Hazards, Exposure Limits
18.3 Table 3: QC Frequency, Criteria and Recommended Corrective Action (ICP-AES)
18.4 Table 4: In house control charted limits for CRI standard
18.5 Appendix A: Standard Preparation Tables
18.6 Appendix B: Terms and Definitions

Table 1: Target Analyte List, Reporting Limit and Wavelengths Used by Instrument

Element	CAS No.	Reporting Limit		Wavelength	
		Water (ug/L)	Soil (mg/Kg)	TJA Trace ICP4	TJA Trace ICP6
Aluminum	7429-90-5	200	20	308.215	308.215
Antimony	7440-36-0	60	6	206.838	206.838
Arsenic	7440-38-2	10	1.0	189.042	189.042
Barium	7440-39-3	200	20	493.409	493.409
Beryllium	7440-41-7	5	0.5	313.042	313.042
Cadmium	7440-43-9	5	0.5	226.502	226.502
Calcium	7440-70-2	5000	500	317.933	317.933
Chromium	7440-47-3	10	1	267.716	267.716
Cobalt	7440-48-4	50	5	228.616	228.616
Copper	7440-50-8	25	2.5	324.754	324.753
Iron	7439-89-6	200	20	271.441	271.441
Lead	7439-92-1	10	1.0	220.353	220.353
Magnesium	7439-95-4	5000	500	279.078	279.078
Manganese	7439-96-5	15	1.5	257.610	257.610
Nickel	7440-02-0	40	4	231.604	202.030
Potassium	7440-09-7	5000	500	766.491	766.491
Selenium	7782-49-2	35	3.5	196.026	196.026
Silver	7440-22-4	10	1	328.068	328.068
Sodium	7440-23-5	5000	500	330.232	330.232
Thallium	7440-28-0	25	2.5	190.864	190.864
Vanadium	7440-62-2	50	5	292.402	292.402
Zinc	7440-66-6	20	2	231.856	206.200
Boron	7440-42-8	100	10	249.678	246.678
Molybdenum	7439-98-7	10	1	202.030	202.030
Tin	7440-31-5	20	2	189.989	189.989
Silicon	7440-21-3	100	10	288.158	--
Titanium	7440-32-6	20	2	--	334.941
Strontium	7440-24-6	20	2	--	421.552
Phosphorous	7723-14-0	250	25	--	178.287

Table 2: Primary Materials Used, Hazards, Exposure Limits

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 3: QC Frequency, Criteria and Recommended Corrective Action (ICP-AES)

QC Check	Acronym	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	ICAL	Daily	NA	NA
Initial Calibration Verification	ICV	After each calibration, prior to sample analysis.	±10% of expected value %RSD between replicate integrations <5%	Correct problem, verify second source standard. If that fails, repeat calibration.
Initial Calibration Blank	ICB	Beginning of analytical sequence after ICV	No analytes ≥ RL DoD: No analytes >2X MDL	Correct problem and reanalyze
Continuing Calibration Verification	CCV	At beginning of analytical sequence, after every 10 samples and at the end of the analytical sequence	±10% of expected value %RSD between replicate integrations <5%	Correct problem, reanalyze CCV. If that fails, repeat calibration and reanalyze all samples since last successful calibration.
Calibration Blank	CCB	Beginning of sample run, after every 10 samples and at end of the sequence (i.e. after each CCV)	No analytes ≥ RL DoD: No analytes > 2XMDL	Correct problem and reanalyze the calibration blank and previous 10 samples.
Interference Check Solutions	ICSA ICSAB	At the beginning of the analytical run	±20% of expected value	Stop analysis, locate and correct problem, reanalyze ICS and all associated QC and samples.
Low Level Standard	CRI	Daily, after ICSA and ICSAB	For DoD work: ±30%	Examine project DQO's. If necessary, reanalyze.
Method Blank	MB	One per digestion batch	No analytes ≥RL DoD: No analytes > 2XMDL	Correct problem, redigest and reanalyze MB and associated samples.
Laboratory Control Sample	LCS	One per digestion batch *A duplicate LCS (LCSD) should be performed only per client request.*	%R= 80-120	Correct problem, redigest and reanalyze LCS, MB and associated samples for failed analytes if sufficient sample volume is available.
Matrix Spike	MS	One per batch of twenty samples or less	%R= 80-120	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Sample Duplicate	SD	One per batch of twenty samples or less	RPD ≤ 20	Examine project DQO's with Project Manager. Evaluate data to determine source of difference between results
Linear Range Verification	LRV	Once per analytical batch (DoD work only)	%R= 90-110	Stop analysis, correct problem and re-analyze all samples with concentrations that exceed respective calibration standard level.
Serial Dilution	L	Each digestion batch	5X dilution within ±10% of original sample result	Perform Post Digestion Spike Flag Data
Post Digestion Spike	A	When dilution test fails or analyte concentration in all samples <25 x MDL	%R within 75-125	Flag data

Table 4: LRV solution: DoD only

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Aluminum (AL)	10000 ppm	60 mL	1000 mL	600000 ug/L
Antimony (Sb)	1000 ppm	4.0 mL		4000 ug/L
Arsenic (As)	1000 ppm	2.0 mL		2000 ug/L
Barium (Ba)	1000 ppm	4.0 mL		4000 ug/L
Beryllium (Be)	1000 ppm	2.0 mL		2000 ug/L
Cadmium (Cd)	1000 ppm	2.0 mL		2000 ug/L
Calcium (Ca)	10000 ppm	60 mL		600000 ug/L
Chromium (Cr)	1000 ppm	4.0 mL		4000 ug/L
Cobalt (Co)	1000 ppm	4.0 mL		4000 ug/L
Copper (Cu)	1000 ppm	4.0 mL		4000 ug/L
Iron (Fe)	10000 ppm	30 mL		300000 ug/L
Lead (Pb)	1000 ppm	4.0 mL		4000 ug/L
Magnesium (Mg)	10000 ppm	60 mL		600000 ug/L
Manganese (Mn)	1000 ppm	4.0 mL		4000 ug/L
Nickel (Ni)	1000 ppm	4.0 mL		4000 ug/L
Potassium (K)	10000 ppm	10 mL		100000 ug/L
Selenium (Se)	1000 ppm	2.0 mL		2000 ug/L
Sodium (Na)	10000 ppm	10 mL		100000 ug/L
Thallium (Tl)	1000 ppm	2.0 mL		2000 ug/L
Vanadium (V)	1000 ppm	4.0 mL		4000 ug/L
Zinc (Zn)	1000 ppm	4.0 mL		4000 ug/L

Solution: *5% HCL / 2% HN03*

Appendix A: Standard Preparation Tables

The formulations provide in this Appendix are recommended. When concentration of the component standard changes then the formulation and final concentrations must also be adjusted. Unless otherwise noted all standards are prepared in a solutions that consists of 5% Hydrochloric Acid and 2% Nitric Acid. Unless otherwise noted primary source standards are purchased from SPEX and second source standards are purchased from Inorganic Ventures.

Calibration Standard #7

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Aluminum (Al)	500 ppm	200 mL of X-AQU-4	2000 mL	50000 ug/L
Calcium (Ca)	500 ppm			50000 ug/L
Iron (Fe)	500 ppm			50000 ug/L
Magnesium (Mg)	500 ppm			50000 ug/L
Sodium (Na)	500 ppm			50000 ug/L
Potassium (K)	500 ppm			50000 ug/L

Calibration Standard #8

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Arsenic (As)	1000 ppm	1.0 mL	2000 mL	500 ug/L
Antimony (Sb)	1000 ppm	1.0 mL		500 ug/L
Lead (Pb)	1000 ppm	2.0 mL		1000 ug/L
Selenium (Se)	1000 ppm	1.0 mL		500 ug/L
Strontium (Sr)	1000 ppm	2.0 mL		1000 ug/L
Thallium (Tl)	1000 ppm	1.0 mL		500 ug/L
Tin (Sn)	1000 ppm	2.0 mL		1000 ug/L
Titanium (Ti)	1000 ppm	2.0 mL		1000 ug/L

Calibration Standard #4

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Silver (Ag)	1000 ppm	1.0 mL	2000	500 ug/L
Phosphorus (P)	1000 ppm	2.0 mL		1000 ug/L
Silicon (Si)	1000 ppm	10 mL		5000 ug/L
Aluminum (Al)	100 ppm	20 mL of X-AQU-5		1000 ug/L
Antimony	100 ppm			1000 ug/L
Barium	100 ppm			1000 ug/L
Berylium (Be)	50 ppm			500 ug/L
Boron (B)	100 ppm			1000 ug/L
Cadmium (Cd)	50 ppm			500 ug/L
Chromium (Cr)	100 ppm			1000 ug/L
Cobalt (Co)	100 ppm			1000 ug/L
Copper (Cu)	100 ppm			1000 ug/L
Iron (Fe)	100 ppm			1000 ug/L
Lead (Pb)	100 ppm			1000 ug/L
Magnesium (Mg)	100 ppm			1000 ug/L
Manganese (Mn)	100 ppm			1000 ug/L
Molybdenum (Mo)	100 ppm			1000 ug/L
Nickel (Ni)	100 ppm			1000 ug/L
Potassium (K)	100 ppm			1000 ug/L
Sodium (Na)	100 ppm			1000 ug/L
Strontium (Sr)	50 ppm			500 ug/L
Titanium (Ti)	100 ppm			1000 ug/L
Vanadium (V)	100 ppm			1000 ug/L
Zinc (Zn)	100 ppm			1000 ug/L

CRI Intermediate Solution #1

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Antimony (Sb)	1000 ppm	6.0 mL	100 mL	60000 ug/L
Arsenic (As)	1000 ppm	1.0 mL		10000 ug/L
Berylium (Be)	1000 ppm	0.5 mL		5000 ug/L
Cadmium (Cd)	1000 ppm	0.5 mL		5000 ug/L
Chromium (Cr)	1000 ppm	1.0 mL		10000 ug/L
Cobalt (Co)	1000 ppm	5.0 mL		50000 ug/L
Copper (Cu)	1000 ppm	2.5 mL		25000 ug/L
Lead (Pb)	1000 ppm	1.0 mL		10000 ug/L
Mangenese (Mn)	1000 ppm	1.5 mL		15000 ug/L
Nickel (Ni)	1000 ppm	4.0 mL		40000 ug/L
Selenium (Se)	1000 ppm	3.5 mL		35000 ug/L
Silver (Ag)	1000 ppm	1.0 mL		10000 ug/L
Thallium (Tl)	1000 ppm	2.5 mL		25000 ug/L
Vanadium (V)	1000 ppm	5.0 mL		50000 ug/L

Solution: *5% HCL / 2% HN03*

CRI Intermediate Solution #2

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Aluminum (Al)	10000 ppm	0.2 mL	100 mL	20000 ug/L
Barium (Ba)	1000 ppm	2.0 mL		200000 ug/L
Calcium (Ca)	10000 ppm	5.0 mL		500000 ug/L
Iron (Fe)	10000 ppm	0.2 mL		20000 ug/L
Potassium (K)	10000 ppm	5.0 mL		500000 ug/L
Magnesium (Mg)	10000 ppm	5.0 mL		500000 ug/L
Sodium (Na)	10000 ppm	5.0 mL		500000 ug/L
Boron (B)	1000 ppm	1.0 mL		10000 ug/L
Molybdenum (Mo)	1000 ppm	0.1 mL		1000 ug/L
Phosphorus (P)	1000 ppm	2.5 mL		25000 ug/L
Silicon (Si)	1000 ppm	1.0 mL		10000 ug/L
Strontium (Sr)	1000 ppm	0.2 mL		2000 ug/L
Tin (Sn)	1000 ppm	0.2 mL		2000 ug/L
Titanium (Ti)	1000 ppm	0.2 mL		2000 ug/L

Solution: *5% HCL / 2% HN03*

CRI Intermediate Solution #3

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Zinc (Zn)	1000 ppm	2.0 mL	100 mL	20000 ug/L

Solution: *5% HCL / 2% HN03*

CRI Working Standard Solution

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Antimony (Sb)	60000 ug/L	1.0 mL of CRI #1	100 mL	60 ug/L
Arsenic (As)	10000 ug/L			10 ug/L
Berylium (Be)	5000 ug/L			5 ug/L
Cadmium (Cd)	5000 ug/L			5 ug/L
Chromium (Cr)	10000 ug/L			10 ug/L
Cobalt (Co)	50000 ug/L			50 ug/L
Copper (Cu)	25000 ug/L			25 ug/L
Lead (Pb)	10000 ug/L			10 ug/L
Mangenese (Mn)	15000 ug/L			15 ug/L
Nickel (Ni)	40000 ug/L			40 ug/L
Selenium (Se)	35000 ug/L			35 ug/L
Silver (Ag)	10000 ug/L			10 ug/L
Thallium (Tl)	25000 ug/L			25 ug/L
Vanadium (V)	50000 ug/L			50 ug/L
Aluminum (Al)	20000 ug/L	10 mL of CRI #2		200 ug/L
Barium (Ba)	20000 ug/L			200 ug/L
Calcium (Ca)	500000 ug/L			5000 ug/L
Iron (Fe)	20000 ug/L			200 ug/L
Potassium (K)	500000 ug/L			5000 ug/L
Magnesium (Mg)	500000 ug/L			5000 ug/L
Sodium (Na)	500000 ug/L			5000 ug/L
Boron (B)	10000 ug/L			100 ug/L
Molybdenum (Mo)	1000 ug/L			10 ug/L
Phosphorus (P)	25000 ug/L			250 ug/L
Silicon (Si)	10000 ug/L			100 ug/L
Strontium (Sr)	2000 ug/L			20 ug/L
Tin (Sn)	2000 ug/L			20 ug/L
Titanium (Ti)	2000 ug/L			20 ug/L
Zinc (Zn)	20000 ug/L	1.0 mL of CRI #3		20 ug/L

Solution: *5% HCL / 2% HN03*

ICSA Working Standard Solution

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Aluminum (AL)	10000 ppm	100 mL	2000 mL	500000 ug/L
Calcium (Ca)	10000 ppm	100 mL		500000 ug/L
Iron (Fe)	10000 ppm	40 mL		200000 ug/L
Magnesium (Mg)	10000 ppm	100 mL		500000 ug/L

Solution: *5% HCL / 2% HN03*

ICSAB Working Standard Solution

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Silver (Ag)	1000 ppm	0.40 mL	2000 mL	200 ug/L
Antimony (Sb)	1000 ppm	1.2 mL		600 ug/L
Zinc (Zn)	1000 ppm	2.0 mL		1000 ug/L
Cadmium (Cd)	1000 ppm	2.0 mL		1000 ug/L
Nickel (Ni)	1000 ppm	2.0 mL		1000 ug/L
Arsenic (As)	1000 ppm	0.20 mL		100 ug/L
Boron (B)	1000 ppm	3.0 mL		1500 ug/L
Tin (Sn)	1000 ppm	3.0 mL		1500 ug/L
Iron (Fe)	10000 ppm	40 mL		200000 ug/L
Calcium (Ca)	10000 ppm	100 mL		500000 ug/L
Magnesium (Mg)	10000 ppm	100 mL		500000 ug/L
Aluminum (Al)	10000 ppm	100 mL		500000 ug/L
Molybdenum (Mo)	1000 ppm	2.0 mL		1000 ug/L
Silicon (Si)	1000 ppm	2.0 mL		1000 ug/L
Selenium (Se)	1000 ppm	0.10 mL		50 ug/L
Thallium (Tl)	1000 ppm	0.20 mL		100 ug/L
Barium (Ba)	1000 ppm	1.0 mL		500 ug/L
Berylium (Be)	1000 ppm	1.0 mL		500 ug/L
Cobalt (Co)	1000 ppm	1.0 mL		500 ug/L
Chromium (Cr)	1000 ppm	1.0 mL		500 ug/L
Copper (Cu)	1000 ppm	1.0 mL		500 ug/L
Manganese (Mn)	1000 ppm	1.0 mL		500 ug/L
Lead (Pb)	1000 ppm	0.10 mL		50 ug/L
Vanadium (V)	1000 ppm	1.0 mL		500 ug/L
Phosphorus (P)	1000 ppm	1.0 mL		500 ug/L
Titanium (Ti)	1000 ppm	1.0 mL		500 ug/L
Strontium (Sr)	1000 ppm	0.50 mL		250 ug/L

Solution: *5% HCL / 2% HN0*

Initial Calibration Verification (ICV)

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Aluminum (Al)*	100 ppm	20 mL of AT-2	2000 mL	1000 ppb
Lead (Pb)	100 ppm			1000 ppb
Barium (Ba)	50 ppm			500 ppb
Berylium (Be)	50 ppm			500 ppb
Boron (B)	50 ppm			500 ppb
Cadmium (Cd)	50 ppm			500 ppb
Chromium (Cr)	50 ppm			500 ppb
Cobalt (Co)	50 ppm			500 ppb
Iron (Fe)*	50 ppm			500 ppb
Manganese (Mn)	50 ppm			500 ppb
Nickel (Ni)	50 ppm			500 ppb
Silver (Ag)	50 ppm			500 ppb
Strontium (Sr)	50 ppm			500 ppb
Vanadium (V)	50 ppm			500 ppb
Zinc (Zn)	50 ppm			500 ppb
Aluminum (Al)*	500 ppm	100 mL of AT-3		25000 ppb
Calcium (Ca)	500 ppm			25000 ppb
Iron (Fe)*	500 ppm			25000 ppb
Magnesium (Mg)	500 ppm			25000 ppb
Potasium (K)	500 ppm			25000 ppb
Sodium (Na)	500 ppm			25000 ppb
Arsenic (As)	1000 ppm	0.50 mL		250 ppb
Antimony (Sb)	1000 ppm	0.50 mL		250 ppb
Molybdenum (Mo)	1000 ppm	1.0 mL		500 ppb
Phosphorous (P)	1000 ppm	1.0 mL		500 ppb
Selenium (Se)	1000 ppm	0.50 mL		250 ppb
Silicon (Si)	1000 ppm	0.50 mL		250 ppb
Tin (Sn)	1000 ppm	0.50 mL		250 ppb
Thallium (Tl)	1000 ppm	0.50 mL		250 ppb
Titanium (Ti)	1000 ppm	1.0 mL		500 ppb

*Elements present in multiple intermediate solutions
Solution: *5% HCL / 2% HN03*

CCV Working Standard Solution

Element	Stock Standard	Volume Used	Final Volume	Final Concentration
Aluminum (Al)*	50000 ppb	600 mL of STD 7	1000 mL	30000 ug/L
Calcium (Ca)	50000 ppb			30000 ug/L
Iron (Fe)*	50000 ppb			30000 ug/L
Magnesium (Mg)*	50000 ppb			30000 ug/L
Sodium (Na)*	50000 ppb			30000 ug/L
Potassium (K)*	50000 ppb			30000 ug/L
Arsenic (As)	500 ppb	200 mL of STD 8		100 ug/L
Antimony (Sb)*	500 ppb			100 ug/L
Lead (Pb)*	1000 ppb			200 ug/L
Selenium (Se)	500 ppb			100 ug/L
Strontium (Sr)*	1000 ppb			200 ug/L
Thallium (Tl)	500 ppb			100 ug/L
Tin (Sn)	1000 ppb			200 ug/L
Titanium (Ti)*	1000 ppb			200 ug/L
Aluminum (Al)*	1000 ppb	200 mL of STD 4		200 ug/L
Antimony (Sb)*	1000 ppb			200 ug/L
Barium (Ba)	1000 ppb			200 ug/L
Berylium (Be)	500 ppb			100 ug/L
Boron (B)*	1000 ppb			200 ug/L
Cadmium (Cd)	500 ppb			100 ug/L
Chromium (Cr)	1000 ppb			200 ug/L
Cobalt (Co)	1000 ppb			200 ug/L
Copper (Cu)	1000 ppb			200 ug/L
Iron (Fe)*	1000 ppb			200 ug/L
Lead (Pb)*	1000 ppb			200 ug/L
Magnesium (Mg)*	1000 ppb			200 ug/L
Manganese (Mn)	1000 ppb			200 ug/L
Molybdenum (Mo)	1000 ppb			200 ug/L
Nickel (Ni)	1000 ppb			200 ug/L
Phosphorous (P)	1000 ppb			200 ug/L
Potassium (K)*	1000 ppb			200 ug/L
Silicon(Si)	5000 ppb			1000 ug/L
Silver (Ag)	500 ppb			100 ug/L
Sodium (Na)*	1000 ppb			200 ug/L
Strontium (Sr)*	500 ppb			100 ug/L
Titanium (Ti)*	1000 ppb			200 ug/L
Vanadium (V)	1000 ppb			200 ug/L
Zinc (Zn)	1000 ppb			200 ug/L
Boron (B)*	1000 ppm	0.50 mL		500 ug/L

*Elements present in multiple intermediate solutions

Solution: *5% HCL / 2% HN03*

Appendix B: Terms & Definitions

Analyte: The element or ion an analysis seeks to determine; the element of interest.

Analytical Sequence: The actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final CCV or CCB.

Background Correction: A technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Calibration Blank: A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standards: A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Low Level Standard: A single parameter or multi-parameter standard solution prepared at the CRQL and used to verify the instrument calibration at low levels.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Dissolved Metals: Analyte elements in a water/aqueous sample which will pass through a 0.45 micrometer (µm) filter.

Dry Weight: The weight of a sample based on percent solids. The weight after drying in an oven.

Duplicate: A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES): A technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of

the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): solution prepared from a separate source from that which is used to prepare the calibration curve.

Interferents: Substances, which affect the analysis for the element of interest.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Linear Range, Linear Dynamic Range: The concentration range over which the instrument response remains linear.

Matrix: The predominant material which the sample to be analyzed is composed of.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Effect: In general, the effect of particular matrix constituents.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Percent Difference (%D): The difference between the two values divided by one of the values.

Percent Solids (%S): The proportion of solid in a soil sample determined by drying an aliquot of the sample.

Preparation Blank: An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Relative Percent Difference (RPD): The relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Sample: A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Serial Dilution: The dilution of a sample by a factor of five. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

STANDARD OPERATING PROCEDURE
STL BURLINGTON

MERCURY IN NONPOTABLE WATER (COLD VAPOR TECHNIQUE)
SW-846 7470A

Applicable Matrix: Nonpotable Water

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground water using a cold vapor technique with atomic absorption (AA).

1.2 The reporting limit is 0.2 ug/L.

2.0 SUMMARY OF METHOD

2.1 An aliquot of sample is acid digested with potassium permanganate and potassium persulfate for two hours in a water bath maintained at a temperature of 95°C. After digestion, hydroxylamine hydrochloride is added to reduce excess permanganate. The digestate is placed on a closed-system mercury autoanalyzer and stannous chloride is added to each sample. The elemental mercury released is measured spectrophotometrically at a wavelength of 253.7 nm. Absorbance (peak height) is measured as a function of mercury concentration and sample results are calculated from the response of the sample absorbance applied against the calibration curve.

2.2 This procedure is based on SW-846 Method 7470A “Mercury in Liquid Waste (Manual Cold-Vapor Technique), Revision 1, September 1994.

3.0 DEFINITIONS

3.1 Definitions are included in Appendix A.

4.0 INTERFERENCES

4.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

4.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

5.0 SAFETY

5.1 Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2 Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added. Protective clothing such as a lab coat, safety glasses and latex gloves must be worn while performing this procedure.

5.3 Primary Materials Used

Table 1, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 Mercury Auto-Analyzer: Leeman Labs PS 200 and Leeman Labs Hydra AA.

6.2 Block Digester: Environmental Express or equivalent: able to maintain digestates at a temperature of 95°C.

6.3 Polyethylene Digestion Vessels with Volumetric Indicators.

6.4 Volumetric Pipettes: Calibrated daily.

6.5 Top Loading Balance: With capability to measure to 0.1 mg.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent Water

Nitric Acid (HNO_3): concentrated, reagent grade: J.T. Baker or equivalent.

Hydrochloric Acid (HCl): concentrated, reagent grade: J.T. Baker or equivalent.

Stannous Chloride: reagent grade: J.T. Baker or equivalent

Potassium Permanganate ($KMNO_4$): reagent grade: J.T. Baker or equivalent

Potassium Persulfate ($K_2S_2O_8$): reagent grade: J.T. Baker or equivalent

HCl Solution (10% v/v): Add 100 mL of concentrated HCl to a 1 L volumetric flask containing ~800 mL of reagent water. Bring final volume of solution up to 1 L with reagent water.

HNO_3 (50% v/v): Add 10 L of concentrated HNO_3 to 20 L carboy containing 10 L of reagent water.

Stannous Chloride Solution: Add 100 g of $SnCl_2 \cdot H_2O$ to 1 L of 10% hydrochloric acid.

Hydroxylamine Hydrochloride Solution: Dissolve 240 g of Hydroxylamine Hydrochloride in 2 L of reagent water.

Potassium Permanganate Solution ($KMNO_4$) (5% w/v): Dissolve 100g of $KMNO_4$ in 2 L of reagent water.

Potassium Persulfate Solution ($K_2S_2O_8$) (5% w/v): Dissolve 100 g of $K_2S_2O_8$ in 2 L of reagent water.

7.2 Standards

Hg Stock Standard Solution (1000 mg/L): purchase from Spex.

Mercury Intermediate Standard (10,000 ug/L): Add 1 mL of 1000 mg/L Hg Stock Standard Solution and 0.15 mL of concentrated HNO_3 to a 100 mL volumetric flask that contains approximately 80 mL reagent water. Adjust to volume with reagent water. Assign an expiration date of six months from the date made, or the manufacturers date, whichever is sooner.

Mercury Working Standard (100 ug/L): Add 1.0 mL of the Hg Intermediate Standard Solution and .15 mL of concentrated HNO_3 to a 100 mL volumetric flask that contains approximately 80 mL reagent water. Adjust to volume with reagent water. Use this standard to prepare the calibration standards (ICAL & CCV). Prepare this standard each day of use.

ICV Stock Standard Solution (1000 mg/L): purchase from Inorganic Ventures.

ICV Intermediate Standard Solution (10,000 ug/L): Add 1 mL of the 1000 mg/L ICV Stock Standard Solution and 0.15 mL of concentrated HNO_3 to a 100 mL volumetric flask that contains approximately 80 mL reagent water. Adjust to volume with reagent water. Assign an expiration date of six months from the date made, or the manufacturers date, whichever is sooner.

ICV Working Standard Solution (60 ug/L): Add 3 mL of the ICV Intermediate Standard Solution and 0.75 mL of concentrated HNO_3 into a 500 mL volumetric flask that contains approximately 300 mL reagent water. Adjust to volume with reagent water. Assign an expiration date of six months from the date made, or the manufacturers date, whichever is sooner.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 A minimum sample volume of 100 mL should be collected in glass or polyethylene containers a sufficient amount of 50% nitric acid solution to acidy the sample to a pH <2. Preservation may be performed in the field or on receipt in the laboratory. Following acidification, the sample should not be processed for at least 16 hours. The pH of the sample must be checked prior to withdrawing an aliquot for digestion. If the pH of the

sample is >2, more acid must be added and the sample held for an additional 16 hours until the pH is verified as less than 2.

8.2 The holding time is 28 days from collection.

8.3 Unless otherwise specified by client or regulatory program, after analysis, samples are retained for a minimum of 60 days and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 QC Requirements

The following QC samples are analyzed with each digestion batch: Method Blank (MB) Laboratory Control Sample (LCS), a Matrix Spike (MS), and a Sample Duplicate (SD).

In addition to calibration (ICAL), instrument standardization is checked with the following QC samples, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), and Continuing Calibration Blank (CCB). A low level standard (CRI) is analyzed per client request. Sample results that exceed the range of calibration are diluted and reanalyzed such that the diluted sample result are near the midpoint or in the upper half of the calibration range.

The minimum frequency requirements, acceptance criteria and recommended corrective action for QC samples are summarized in Table 2, Section 18.0.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibration

Calibrate the autoanalyzers daily with five calibration standards and a blank using the instrument operating conditions established by the instrument manufacturer. Prepare the calibration standards daily by making successive dilutions of the Hg Working Standard Solution (100 ug/L) in appropriate volume of reagent water to achieve a final volume of 50 mL. Refer to Table 3, Section 18.0 for standard formulation.

Process the calibration standards following the procedures given in Section 11.1. The instrument's data system constructs a standard curve by plotting the instrument response from each standard solution against the final concentration and using linear regression, the data system calculates the correlation coefficient. The correlation coefficient must be greater than or equal to 0.995.

10.2 Initial Calibration Verification (ICV)

Following calibration, analyze the ICV. The ICV is a second source standard whose concentration (3 ug/L) is near the midpoint of the calibration range but at a different

concentration than the CCV (5 ug/L). Process the ICV following the procedure given in Section 11.1. The percent recovery of the ICV must be within 90-110%.

10.3 Continuing Calibration Verification (CCV).

Analyze a CCV following the ICB, after every 10th sample and at the end of the sample run. The CCV standard is at a concentration of 5ug/L and is prepared from the same source of standard used for the calibration standards. Process the CCV following the procedure given in Section 11.1. The percent recovery of the CCV must be 80-120%.

10.4 Initial Calibration Blanks (ICB) and Continuing Calibration Blanks (CCB)

Analyze an ICB after the ICV. Analyze a CCB after each CCV. The results of each calibration blank must be less than the RL.

10.5 Support Equipment Calibration

Check the calibration of the auto-pipettes and the top-loading balance on the day of use prior to use and record the calibration check in the logbook designated for this purpose.

11.0 PROCEDURE

11.1 Sample, QC & Standard Preparation

Transfer a 50 mL aliquot of sample to a labeled polyethylene digestion vessel. Use 50 mL of reagent water for the method blank, LCS and each calibration blank. Add 0.5 mL of the Hg working standard solution (100 ug/L) to the LCS and the matrix spike.

To prepare the ICV, transfer 5 mL of the ICV working standard solution (30 ug/L) to a labeled polyethylene digestion vessel and add 45 mL of reagent water.

For each CCV, transfer 2.5 mL of the Hg working standard solution (100 ug/L) to a labeled polyethylene digestion vessel and add 47.5 mL of reagent water.

If analysis of a low-level standard is requested, transfer 0.1 mL of the Hg working standard solution (100ug/L) to a labeled polyethylene digestion vessel and adjust the volume to 50 mL with reagent water.

11.2 Digestion

Add 2.5 mL of concentrated sulfuric acid and 1.25 mL of concentrated nitric acid to each digestion bottle, mixing after each addition. Add 7.5 mL of potassium permanganate solution to each digestion vessel and allow the vessels to stand for 15 minutes. During this time the solution should turn and remain the color purple. If after 15 minutes, the solution does not remain purple, add more potassium permanganate (~2.5 mL aliquots) and ensure that equal amounts of potassium permanganate are added to the standards and blanks.

Add 4 mL of potassium persulfate to each vessel and heat for two hours in a block digester maintained at 95°C. Cool and add 3 mL of hydroxylamine hydrochloride to each vessel to reduce the excess permanganate. Transfer each digestate to individual autoanalyzer tubes. If additional potassium permanganate was added prior to digestion, additional aliquots of hydroxylamine hydrochloride will be needed

11.3 Instrument Set Up & Analysis

Turn the instrument lamp, gas and pump on and allow 15 minutes for the instrument to warm up. Fill the rinse bath with 10% hydrochloric acid solution. Check all tubing connections and reset the calibration curve. Check the stannous chloride reductant reservoir and fill if necessary.

Select the autosampler template and enter the sample Ids in the order of analysis. Place the samples, calibration blanks, calibration standards, and performance check standards in the position on the autosampler rack that corresponds to their assigned position in the autosampler template. Place the autosampler rack in the autosampler tray and initiate the software macro to begin analysis. An example analytical sequence is given below:

Example Analytical Sequence:
Calibration Blank
0.2 Calibration Standard
0.5 Calibration Standard
1.0 Calibration Standard
5.0 Calibration Standard
10.0 Calibration Standard
ICV
ICB
CRI (if client requested)
CCV
CCB
10 Samples*
CCV
CCB
10 Samples*
CCV
CCB

**The number of samples between each CCB/CCV (10) includes the method blank, laboratory control sample, matrix spikes, and sample duplicates.*

During analysis, the data processing system constructs a calibration curve by plotting the absorbence of standards versus units of mercury and sample concentrations are determined from the calibration curve.

After analysis is complete, review the data against the acceptance criteria given in Table 2, Section 18.2 and perform corrective action as needed. Dilute and reanalyze any samples that exceed the linear range.

12.0 CALCULATIONS

12.1 Sample Concentration

$$C_{(ug/L)} = \frac{\mu g}{L_{dig}} * \frac{V_{dig}}{V_{samp}}$$

Where:
$\mu g/L_{dig}$ = Instrument result adjusted for dilution factors
V_{dig} = Final digestate volume in liters
V_{samp} = Sample volume in liters

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Review the samples, standards and QC samples against the acceptance criteria in Table 2, Section 18.2. If the results do not fall within the established limits or criteria, corrective action is required as contained in Table 2. Recommended corrective action is provided in Table 2. If corrective action is not taken or unsuccessful, the situation should be documented and reported in the project narrative. All data that does not meet established criteria must be noted in the project narrative.

14.0 METHOD PERFORMANCE

14.1 A demonstration of analyst capability (IDOC) is required prior to use of this SOP and any time there is a significant change in instrument type, personnel or test method. IDOC procedures are further described in the laboratory SOP for employee training.

14.2 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in the laboratory SOP for method detection limit studies.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 The following waste streams are produced when this method is carried out.

- Acid Waste - Prep - 55 gallon drum.

- Acid Waste - Analysis – 5 gallon carboy.

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

16.1 SW-846 Method 7470A "Mercury in Liquid Waste (Manual Cold-Vapor Technique)", Revision 1, September 1994.

17.0 REVISION HISTORY

17.1 Section 7.1: The expiration date of the 100 ug/L working standard was changed from 6 months to daily.

17.2 Sections 10.0 and 11.0: Updated to reflect a sample volume of 50 mL instead of 100 mL.

17.3 Table 2, Section 18.2: Frequency for matrix spike was changed from per client request to a frequency of 1 per batch of 20 or fewer samples. The acceptance criteria for the matrix spike was changed from 75-125 to 85-115 in order to be consistent with the criteria for the LCS. Control limits based on control charts were added for the low-level standard (CRI).

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Primary Materials Used, Hazards & Exposure Limits
18.2 Table 2: QC Summary and Recommended Corrective Action
18.3 Table 3: Calibration Standards
18.4 Appendix A: Terms and Definitions

Table 1: Primary Materials Used, Hazards & Exposure Limit

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous: it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 2: QC Frequency, Criteria and Recommended Corrective Action (SW-846 7470A)

QC Check	Acronym	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	ICAL	Daily	r ≥ 0.995	Correct problem and repeat calibration
Initial Calibration Verification	ICV	After each calibration, prior to sample analysis.	±10% of expected value	Correct problem, verify second source standard. If that fails, repeat calibration.
Initial Calibration Blank	ICB	Beginning of analytical sequence after ICV	No analytes ≥RL DoD: ≥MDL	Correct problem and reanalyze
Low Level Standard	CRI	Client Request	%R (30-131) [1]	Flag outliers
Continuing Calibration Verification	CCV	Beginning of sequence, after every 10 samples and at the end of the analytical sequence	±20% of expected value	Correct problem, reanalyze CCV. If that fails, repeat calibration and reanalyze all samples since last successful calibration.
Calibration Blank	CCB	After every 10 samples and at end of the sequence (i.e. after each IPC)	No analytes ≥RL DoD: ≥MDL	Correct problem and reanalyze the calibration blank and previous 10 samples.
Method Blank	MB	One per digestion batch of 20 or less samples	No analytes ≥RL DoD: ½ ≥RL	Correct problem, redigest and reanalyze MB and associated samples.
Laboratory Control Sample	LCS	One per digestion batch of 20 or less samples	%R (85-115)	Correct problem, redigest and reanalyze LCS, MB and associated samples for failed analytes if sufficient sample volume is available.
Matrix Spike	MS	One per batch of twenty samples or less	%R (85-115)	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Sample Duplicate	SD	One per batch of twenty samples or less	RPD ≤ 20	Examine project DQO's with Project Manager. Evaluate data to determine source of difference between results

[1] *The control limits for the low level standard (CRI) are based on control charts and are subject to change each time control charts are generated.*

Table 3: Calibration Standard Levels and preparation instructions

Level	Hg Standard 100 ug/L (mL)	Level 4	Final Concentration (ug/L)
Blank	0	Level 5	0
Level 1	0.1	50	0.2
Level 2	0.25	50	0.5
Level 3	0.5	50	1
Level 4	2.5	50	5
Level 5	5	50	10

Appendix A: Terms & Definitions

Analyte: The element or ion an analysis seeks to determine: the element of interest.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Calibration: The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards.

Calibration Blank: A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Duplicate: A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): solution prepared from a separate source from that which is used to prepare the calibration curve.

Interferents: Substances which affect the analysis for the element of interest.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Relative Percent Difference (RPD): As used in the SOW and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

Sample: A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

STL BURLINGTON
STANDARD OPERATING PROCEDURE

MERCURY (COLD VAPOR TECHNIQUE)
SW-846 Method 7471A
Applicable Matrix: Solid and Chemical Materials

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the determination of total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials.

1.2 The routine RL for solid samples is 0.033 mg/Kg based on a sample digestion weight of 0.3 grams and a final volume of 50 mL.

2.0 SUMMARY OF METHOD

2.1 A portion of solid sample is acid digested for 2 minutes at a temperature of 95°C then digested with potassium permanganate and potassium persulfate for 30 minutes at a temperature of 95°C. Hydroxylamine hydrochloride is added to each digestate in order to reduce excess permanganate. The digestate is placed on a closed-system mercury autoanalyzer and stannous chloride is added to each sample. The elemental mercury released is measured spectrophotometrically at a wavelength of 253.7 nm. The concentration is calculated from the response of the sample absorbance applied against the calibration curve.

2.2 This procedure is based on SW-846 Method 7471A, Revision 1, September 1994.

3.0 DEFINITIONS

3.1 A list of terms and definitions is given in Appendix A.

4.0 INTERFERENCES

4.1 Potassium permanganate is added to the samples to eliminate possible interference from sulfide. Copper has also been noted as an interferent but per reference method SW-846 7471A concentrations as high as 10mg/Kg had no effect on recovery of mercury from spiked samples.

4.2 Samples high in chlorides may require additional permanganate because during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253nm. Care must be taken to ensure free chlorine is not present and this is accomplished by the addition of hydroxylamine hydrochloride and stannous chloride.

5.0 SAFETY

5.1 Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2 Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added. Protective clothing

such as a lab coat, safety glasses and latex gloves must be worn while performing this procedure.

5.3 Primary Materials Used

Table 1, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 Mercury Auto-Analyzer; Leeman Labs PS 200 and Leeman Labs Hydra AA with Autosampler or equivalent.

6.2 Block Digester, capable of maintaining temperature at 90-95°C: Environmental Express or equivalent.

6.3 Polyethylene Digestion Vessels, 100mL volume with Volumetric Indicators: Environmental Express or equivalent,.

6.4 Volumetric Autopipettes, Range of use 0.2-1.0mL & 1.0-5.0mL: Finpipette or equivalent.

6.5 Top Loading Balance capable of measurements to 0.1mg.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent Water

Aqua Regia: Prepare by carefully adding 3 volumes of concentrate HCl to one volume of concentrated nitric acid.

Nitric Acid (HNO_3), Concentrated: Reagent Grade: J.T. Baker or equivalent.

Hydrocloric Acid (HCl), Concentrated: Reagent Grade: J.T. Baker or equivalent.

Hydroxalimine Hydrochloride: Reagent Grade: J.T. Baker or equivalent.

Potassium Permanganate: Reagent Grade: J.T. Baker or equivalent.

Stannous Chloride Dihydrate Crystal ($SnCl_2$): Reagent Grade, J.T. Baker or equivalent.

HCl (10%): Add 100mL of concentrated HCl to a 1 L volumetric flask containing ~800 mL of reagent water. Adjust to final volume with reagent water.

Stannous Chloride Solution: Add 200 g of $SnCl_2$ to 2 L of 10% hydrochloric acid.

Hydroxylamine Hydrochloride: Dissolve 240 g of Hydroxylamine Hydrochloride in 2 L of reagent water.

Potassium Permanganate ($KMNO_4$): 5% solution w/v: Dissolve 100g of $KMNO_4$ in 2 L of reagent water.

7.2 Standards

Mercury (Hg) Stock Standard Solution (1000mg/L), Spex.

Hg Mercury Intermediate Standard (10,000ug/L): Add 1.0 mL of 1000 mg/L Hg Stock Standard Solution and 0.15 mL of concentrated HNO_3 to a 100 mL volumetric flask that contains approximately 80 mL reagent water. Adjust to volume with reagent water. Assign an expiration date of six months from the date made, or the manufacturers date, whichever is sooner.

Hg Working Standard (100 ug/L): Add 1.0 mL of the Hg Intermediate Standard Solution and 0.15 mL of concentrated HNO_3 to a 100 mL volumetric flask that contains approximately 80 mL reagent water. Adjust to volume with reagent water. Use this standard to prepare the calibration standards (ICAL & CCV). Prepare this standard each day of use.

ICV Stock Standard Solution (1000 mg/L), Inorganic Ventures.

ICV Intermediate Standard Solution (10,000 ug/L): Add 1.0 mL of the 1000 mg/L ICV Stock Standard Solution and 0.15 mL of concentrated HNO_3 to a 100 mL volumetric flask that contains approximately 80 mL reagent water. Adjust to volume with reagent water. Assign an expiration date of six months from the date made, or the manufacturers date, whichever is sooner.

ICV Working Standard Solution (30 ug/L): Add 1.5 mL of the ICV Intermediate Standard Solution and 0.75 mL of concentrated HNO_3 into a 500 mL volumetric flask that contains approximately 300 mL reagent water. Adjust to volume with reagent water. Assign an expiration date of six months from the date made, or the manufacturers date, whichever is sooner.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 Samples should be collected in glass or polyethylene containers. Immediately following collection the samples should be cooled to a temperature of 4°C (±2) and maintained at that temperature until digestion.

8.2 The holding time is 28 days from collection of the sample.

8.3 Unless otherwise specified by client or regulatory program, after digestion and analysis, samples are retained for 60 days and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 QC Requirements

The following QC samples are analyzed with each digestion batch: Method Blank (MB) Laboratory Control Sample (LCS), a Matrix Spike (MS), and a Sample Duplicate (SD). Some clients and/or regulatory programs may require a Matrix Spike Duplicate (MSD) instead of and/or in addition to the sample duplicate.

In addition to calibration (ICAL), instrument standardization is checked with the following QC samples, Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), and Calibration Blanks (ICB/CCB). A low level standard (CRI) is analyzed per client request. Sample results that exceed the range of calibration are diluted and reanalyzed such that the diluted sample result in near the midpoint or in the upper half of the calibration range.

The minimum frequency requirements, acceptance criteria and recommended corrective action for QC samples are summarized in Table 2, Section 18.0.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibration

Calibrate the autoanalyzers daily with five calibration standards and a blank using the instrument operating conditions established by the instrument manufacturer. Prepare the calibration standards daily by making successive dilutions of the Hg Working Standard Solution (100 ug/L) in reagent water to achieve a final volume of 50 mL. The final concentration of the prepared calibration standards is as follows:

Calibration Standards

Level	Hg Standard 100ug/L (mL)	Final Volume (mL)	Final Concentration (ug/L)
Blank	0	50	0
Level 1	0.1	50	0.2
Level 2	0.25	50	0.5
Level 3	0.5	50	1
Level 4	2.5	50	5
Level 5	5	50	10

Process the calibration standards following the procedures given in Section 11.1. The instrument data system constructs a standard curve by plotting the instrument response from each standard solution against the final concentration and using linear regression, the data system calculates the correlation coefficient. The correlation coefficient must be greater than or equal to 0.995.

10.2 Initial Calibration Verification (ICV)

Following calibration, analyze the ICV. The ICV is a second source standard whose concentration (3 ug/L) is near the midpoint of the calibration range but at a different concentration than the CCV (5 ug/L). Process the ICV following the procedure given in Section 11.1. The percent recovery of the ICV must be within 90-110%.

10.3 Continuing Calibration Verification (CCV).

Analyze a CCV following the ICB, after every 10^{th} sample and at the end of the sample run. The CCV standard is at a concentration of 5 ug/L and is prepared from the same source of standard used for the calibration standards. Process the CCV following the procedure given in Section 11.1. The percent recovery of the CCV must be 80-120%.

10.4 Calibration Blanks (ICB/CCB)

Analyze an initial calibration blank (ICB) after the ICV. Analyze a continuing calibration blank (CCB) after each CCV. The results of each calibration blank must be less than the RL.

10.5 Support Equipment Calibration

Check the calibration of the auto-pipettes and the top-loading balance on the day of use prior to use and record the calibration check in the logbook designated for this purpose.

11.0 PROCEDURE

11.1 Sample, QC & Standard Preparation

Weigh 0.3 g of sample into a polyethylene digestion vessel. Add 5 mL of reagent water. Use reagent water for the method blank (PBS), the laboratory control sample (LCS), and each calibration blank. Add 1 mL of the Hg working standard solution (100 ug/L) to the LCS and the matrix spike.

To prepare the ICV, transfer 5 mL of the ICV working standard solution (30 ug/L) to a labeled polyethylene digestion vessel and add 45 mL of reagent water.

For each CCV, transfer 2.5 mL of the Hg working standard solution (100 ug/L) to a labeled polyethylene digestion vessel and add 47.5 mL of reagent water.

If analysis of a low-level standard is requested, transfer 0.1 mL of the Hg working

standard solution (100 ug/L) to a labeled polyethylene digestion vessel and adjust the volume to 50 mL with reagent water.

11.2 Digestion

To each sample, standard, and blank add 2.5 mL of aqua regia. Heat for 2 minutes in a digestion block at 95°C. Allow the samples to cool then add 20 mL of reagent water, 7.5 mL of potassium permanganate, and swirl to mix. Return to the block for 30 minutes. Cool and, when ready to analyze, add 3 mL of hydroxylamine hydrochloride to reduce the excess permanganate. Swirl each vessel to ensure that any soluble residue dissolves back into solution. If the color of any sample is still purple, add hydroxylamine hydrochloride in 3 mL increments until the purple color disappears. Add 25 mL of reagent water to each vessel and transfer the digestate to individual autoanalyzer tubes for analysis.

11.3 Instrument Set Up & Analysis

Turn the instrument lamp, gas and pump on and allow 15 minutes for the instrument to warm up. Fill the rinse bath with 10% hydrochloric acid solution. Check all tubing connections and reset the calibration curve. Check the stannous chloride reductant reservoir and fill if necessary.

Select the autosampler template and enter the sample IDs in the order of analysis. Place the samples, calibration blanks, calibration standards, and performance check standards in the position on the autosampler rack that corresponds to their assigned position in the autosampler template. Place the autosampler rack in the autosampler tray and initiate the software macro to begin analysis. An example analytical sequence is given below:

Example Analytical Sequence:
Calibration Blank
0.2 Calibration Standard
0.5 Calibration Standard
1.0 Calibration Standard
5.0 Calibration Standard
10.0 Calibration Standard
ICV
ICB
CRI (if requested)
CCV
CCB
PBS
LCS
7 Samples*
CCV
CCB
9 Samples*

CCV
CCB

**The number of samples between each CCB/CCV (10) includes the method blank, laboratory control sample, matrix spikes, and sample duplicates.*

During analysis, the data processing system constructs a calibration curve by plotting the absorbances of standards versus units of mercury and sample concentrations are determined from the calibration curve.

After analysis is complete, review the data against the criteria given in Table 2, Section 18.0 and perform corrective action, as needed. Dilute and reanalyze any samples that exceed the linear range.

12.0 CALCULATIONS

12.1 Concentration

$$C_{(mg/Kg\ drywt.)} = \frac{\mu g}{L_{dig}} * \frac{V_{dig}}{g_{samp}} * \frac{100}{\%\ solids}$$

Where:
$\mu g/L_{dig}$ = Instrument result adjusted for dilution factors
V_{dig} = Final digestate volume in liters
g_{samp} = Sample weight in grams
% Solids = Percent solids to nearest 0.1%

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Review the samples, standards and QC samples against the acceptance criteria in Table 2, Section 18.0. If the results do not fall within the established limits or criteria, corrective action is required as contained in Table 2. Recommended corrective action is provided in Table 2. If corrective action is not taken or unsuccessful, the situation should be documented and reported in the project narrative. All data that does not meet established criteria must be noted in the project narrative.

14.0 METHOD PERFORMANCE

14.1 A demonstration of analyst capability (IDOC) is required prior to use of this SOP and any time there is a significant change in instrument type, personnel or test method. IDOC procedures are further described in the laboratory SOP for employee training.

14.2 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in the laboratory SOP for method detection limit studies.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is carried out.

- Acid Waste, Digested Samples, Expired Hg Standards

→ *Satellite Waste Container: 55 Gallon Closed Topped Poly labeled "CORROSIVE"*

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REVISION HISTORY

16.1 Title Page: Changed to reflect current management team.

16.2 Section 6.0; replaced water bath with digestion block

16.3 Section 7.0: Added vendor detail.

16.4 Section 7.1: Removed Potassium Persulfate and Potassium Persulfate, 5% Solution. Added Stannous Chloride Dihydrate Crystal. Stannous Chloride Solution changed Stannouse Chloride Dihydrate Crystal grams used and changed liters of hydrochloric acid used.

16.5 Section 7.2: Changed concentration of ICV Working Standard Solution. Also changed mLs of ICV intermediate standard used for that solution.

16.6 Section 11: Added "if requested" to analytical sequence, changed water bath reference in procedure to block digester. Eliminated redundant sample table entry information (duplicate sentence)

16.7 Section 11.1: Changed all volumes in the second and third paragraph. All cut in half.

16.8 Section 11.2: volume of Hydroxylamine hydrochloride used if color still purple cut in half.

16.9 Section 11.3: Added PBS and LCS to analytical sequence.

16.10 Seciton 15.2: Satellite waste container information.

16.11 Table 2; changed DoD blank criteria to 2X MDL

17.0 REFERENCES

17.1 Method 7471A Mercury in Solid or Semisolid Waste (Manual Cold Vapor Technique), Revision 1, September 1994. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Primary Material Used
18.2 Table 2: QC Summary, Acceptance Criteria, Recommended Corrective Action

Table 1: Primary Materials Used (Mercury / CVAA)

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 2: QC Frequency, Criteria and Recommended Corrective Action (SW-846 7471A)

QC Check	Acronym	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	ICAL	Daily	$r \geq 0.995$	Correct problem and repeat calibration
Initial Calibration Verification	ICV	After each calibration, prior to sample analysis.	±10% of expected value	Correct problem, verify second source standard. If that fails, repeat calibration.
Initial Calibration Blank	ICB	Beginning of analytical sequence after ICV	No analytes ≥RL DoD: ≥2XMDL	Correct problem and reanalyze
Low Level Standard	CRI	Per Client Request	%R (30-131) [1]	Correct problem, then reanalyze
Continuing Calibration Verfication	CCV	Beginning of sequence, after every 10 samples and at the end of the analytical sequence	±20% of expected value	Correct problem, reanalyze CCV. If that fails, repeat calibration and reanalyze all samples since last successful calibration.
Calibration Blank	CCB	After every 10 samples and at end of the sequence (i.e. after each IPC)	No analytes ≥RL DoD: ≥2XMDL	Correct problem and reanalyze the calibration blank and previous 10 samples.
Method Blank	MB	One per digestion batch	No analytes ≥RL DoD: ≥2XMDL	Correct problem, redigest and reanalyze MB and associated samples.
Laboratory Control Sample	LCS	One per digestion batch	%R (85-115)	Correct problem, redigest and reanalyze LCS, MB and associated samples for failed analytes if sufficient sample volume is available.
Matrix Spike	MS	One per batch of twenty samples or less	%R (85-115)	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Matrix Spike Duplicate	MSD	Per client request Arizona: 1 MS/MSD per batch	%R (85-115)	Examine project DQO's with Project Manager. Evaluate data to determine if outage is related to analytical error or matrix effect.
Sample Duplicate	SD	One per batch of twenty samples or less	RPD ≤ 20	Examine project DQO's with Project Manager. Evaluate data to determine source of difference between results

[1] *The control limits for the low level standard (CRI) are based on control charts and are subject to change each time control charts are generated.*

Appendix A: Terms & Definitions

Analyte: The element or ion an analysis seeks to determine; the element of interest.

Analytical Sequence: The actual instrumental analysis of the samples from the time of instrument calibration through the analysis of the final CCV or CCB.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Calibration: The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards.

Calibration Blank: A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Duplicate: A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): solution prepared from a separate source from that which is used to prepare the calibration curve.

Interferents: Substances which affect the analysis for the element of interest.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Relative Percent Difference (RPD): As used in the SOW and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

Sample: A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

ARCADIS BBL

Attachment 1-3

STL Wet Chemistry and
Miscellaneous Analysis SOPs

STANDARD OPERATING PROCEDURE
STL BURLINGTON

THE DETERMINATION OF TOTAL DISSOLVED SOLIDS (TDS)
EPA METHOD 160.1

Applicable Matrix: Nonpotable Water

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the determination of Total Dissolved Solids (TDS) also known as Filterable Residue (Gravimetric, Dried at 180^{o}C) in nonpotable water.

1.2 The routine reporting limit (RL) is 5.0 mg/L based on a 100 mL sample volume.

2.0 SUMMARY OF METHOD

2.1 The sample is filtered through a pre-weighed standard glass fiber filter disk. The subsequent filtrate is evaporated to dryness in a drying oven maintained at a temperature of 105°C and then dried to a constant weight in a drying oven maintained at a temperature of 180°C. The concentration of dissolved solids in sample is determined from the difference of the initial and final weight measurements of the filter flask.

2.2 This SOP is based on EPA Method 160.1, “Residue, Filterable (Gravimetric, Dried at 180°C)”, Methods for Chemical Analysis of Water and Wastes (MCAWW) – EPA/600/4-79-020 – Revised March 1983.

The laboratory procedure is modified from the reference method as follows:

- The laboratory uses a drying oven instead of steam baths to evaporate the samples.

3.0 DEFINITIONS

3.1 Definitions are included in Appendix A.

4.0 INTERFERENCES

4.1 Interferences may be caused by contaminants in the reagent water, reagents, and glassware. To minimize interferences, high purity reagents must be used and all glassware prepared following the procedures described in this SOP.

4.2 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hydroscopic and will require prolonged drying, desiccation and rapid weighing.

4.3 Large quantities of residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. To prevent this, Total residue should be limited to 200 mg. See Section 9.2 for quality control.

5.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.1 Specific Safety Requirements

None

5.2 Primary Materials Used

There are no materials used in this method that have a significant or serious hazard rating **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found Section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 EQUIPMENT AND SUPPLIES

6.1 Glass Fiber Filter Discs, Whatman 934-AH, or equivalent.

Note: Because of the physical nature of glass fiber filters, the absolute pore size cannot be controlled or measured. Terms such as "pore size," "collection efficiencies," and "effective retention" are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above.

6.2 Filtering Apparatus equipped with a reservoir and a coarse (40-60 microns) fritted disc, Millipore or equivalent.

6.3 Suction Flask, 2000 mL.

6.4 Erlenmeyer Flasks, 250 mL side-arm.

6.5 Drying oven, 105°C (± 2).

6.6 Drying oven, 180°C (± 5).

6.7 Desiccator, Desiccant: Change the desiccant when the "pebbles" have changed color from blue to pink.

6.8 Analytical Balance, capable of weight measurements to 0.1 mg.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent water

Sodium Chloride; reagent grade; J.T. Baker or equivalent.

7.2 Standards

Sodium Chloride Solution (50 mg/L): Measure 0.1000 g of sodium chloride (NaCl) that has been baked in a drying oven at a temperature of 105°C (± 2) for 2 hours, into a 2000 mL volumetric flask and adjust to volume with reagent water. Assign an expiration date of 6 months from date prepared and store at room temperature. Use this solution for the LCS.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 A minimum sample volume of 100 mL of sample should be collected in plastic or glass unpreserved containers. Immediately following collection all samples should be cooled to 4˚C (±2) and maintained at that temperature until sample preparation and analysis. If Total Suspended Solids (TSS) by EPA Method 160.2 is also being determined from the same sample, the filtrate generated from that procedure may be used for this procedure.

8.2 The holding time for water samples is 7 days from the date of collection.

8.3 Unless otherwise specified by client or regulatory program, samples are retained in the laboratory for 30 days from date of invoice and then disposed of in accordance with federal, state, and local regulations.

9.0 QUALITY CONTROL

9.1 A Method Blank (MB) and a laboratory control sample (LCS) are prepared with each batch of 20 or less samples. A Matrix Duplicate (DP) is also prepared with each batch of 20 or less samples when sufficient sample volume is available. The acceptance criteria for QC samples are provided in Table 1, Section 18.0 along with recommended corrective actions.

9.2 Total residue should be limited to 200 mg because too much residue can entrap water that will not be driven off during drying. To minimize this interference, the analysis for sample results that exceed 2000 mg/L are repeated with a smaller sample volume.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibrate the analytical balance on each day of use prior to use. Record the check in the logbook designated for this purpose.

11.0 PROCEDURE

11.1 Filter Disk Preparation

Assemble the membrane filter apparatus and attach the vacuum. Place a glass fiber filter disc onto the apparatus. Apply the vacuum at constant pressure and rinse the filter disc with three successive 20 mL volumes of reagent water, collecting the water in a

2000 mL suction flask. Discard the rinsate and remove the filter disk from the apparatus and place the filter disk in a drying oven maintained at a temperature of 180°C. Dry the filter disk for a minimum of one hour. Remove the filter disks from the oven and store in a desiccator at ambient temperature until use.

11.2 Flask Preparation

Dry the number of 250 mL side-arm Erlenmeyer flasks needed for analysis in a drying oven maintained at a temperature of 180°C for at least 2 hours. Store the flasks in a dessicator until time of use or if used directly from the oven, cool the flasks in a desiccator for at least 1-hour prior to use.

Tare the calibrated balance and weigh each flask to the nearest 0.1 mg and record the weight on the bench sheet as Flask WT 1. Re-weigh the flask after 15 minutes has elapsed and record the weight as Flask WT 2. Calculate the difference between the two weights by subtracting the second measurement reading from the first. The difference should be ≤ 0.5 mg (0.0005 g). If it is not, repeat the drying cycle until the ≤ 0.5 mg weight difference is obtained. Place the flasks into a desiccator and store for no longer than 3 days.

11.3 Sample Preparation & Analysis

Assemble the filtering apparatus and turn the vacuum “on”. Shake the sample to mix and measure 100 mL of sample into a graduated cylinder. For the method blank, use 100 mL of reagent water. For the LCS, use 100 mL of the 50 mg/L sodium chloride solution. For the matrix duplicate, use another 100 mL aliquot of the parent field sample. Record the sample volume measured on the benchsheet and indicate if the sample volume is a filtrate generated from the laboratory procedure for TSS*.

If TSS (Non-filterable residue) by EPA 160.2 is also being measured, the filtrate that was generated from the TSS procedure may be used for this procedure. Refer to laboratory SOP LM-WC-160.2, Section 11.1.2 for further guidance.

Pour the sample aliquot through the funnel of the filtering apparatus into a labeled 250 mL side arm flask and rinse the filter disk three times with approximately 10 mL of reagent water. Continue to apply vacuum for ~3 minutes after filtration to remove as much water as possible.

Place the sample flasks into an oven maintained at a temperature of 105°C (±2) until there is no visible liquid left. Following that, place the sample flasks into an oven maintained at 180°C (±5) for a minimum of 2 hours. Record the time in and out of the drying ovens on the benchsheet. Remove the sample flasks from the oven and cool in a desiccator.

Tare the calibrated balance and weigh each flask to the nearest 0.1 mg. Record the weight on the bench sheet as Final WT 1. Re-weigh each flask after 15 minutes has elapsed and record the weight as Final WT 2. Calculate the difference between the two

weights by subtracting the second measurement reading from the first. The difference should be ≤ 0.5 mg (0.0005 g). If it is not, repeat the drying cycle until the ≤ 0.5 mg weight difference is obtained.

Calculate the concentration of the TDS using the equation given in Section 12.0.

12.0 CALCULATIONS

12.1 TDS Concentration

$$mg/L = \frac{(A - B) * 1{,}000{,}000}{C}$$

Where:
A = Final WT 2 in g
B = Flask WT 2 in g
C = Sample volume used in mL

12.2 LCS Percent Recovery (%R)

$$\%R = \frac{SR}{SA} * 100\%$$

Where:
SR= LCS Result
SA=LCS TV

12.3 Precision (RPD)

$$\%RPD = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} * 100$$

Where:
D1 = Sample result
D2 = Matrix Duplicate (MD) Result

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Review the samples, standards and QC samples against the performance criteria given in Table 1, Section 18.0. If the results do not fall within the established limits perform the recommended corrective action (See Table 1). If corrective action is not taken or unsuccessful, record the situation with a nonconformance report and report and qualify the data as necessary.

14.0 METHOD PERFORMANCE

14.1 Not applicable.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is carried out.

- Contaminated Filter and Filter residue – Satellite Container: 2.5 L Glass General Waste Stream

Transfer the waste stream to the designated satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REVISION HISTORY

16.1 Title page: Updated to reflect current laboratory management team.

16.2 Section 2.0: Added oven temperature modification.

16.3 Section 7.0: Reagent was added.

16.4 Section 9.2: Changed sample result criteria to 2000 mg/L (2000 mg/L = 200 mg/100 mL).

16.5 Section 11.3: added drying step.

16.6 Section 15.2: removed acidic waste as a waste stream.

16.7 Section 16.0: Renamed Revision History.

16.8 Section 17.0: Renamed References.

16.9 Section 18.0: Section Added.

16.10 Entire document: changed evaporating oven criteria to 105°C (±2).

17.0 REFERENCES

17.1 EPA Method 160.1, "Residue, Filterable (Gravimetric, Dried at 180°C)", Methods for Chemical Analysis of Water and Wastes (MCAWW) – EPA/600/4-79-020 – Revised March 1983.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table1: QC Summary and Recommended Corrective Action
18.2 Appendix A: Terms and Definitions

Table 1: QC Summary and Recommended Corrective Action

QC Item	Acronym	Frequency	Acceptance Criteria	Corrective Action
Method Blank	MB	Each batch of 20 samples or less.	< RL < ½ RL (DoD only)	Re-analyze, if still above re-prepare and reanalyze batch.
Laboratory Control Sample	LCS	Each batch of 20 samples or less.	85-115%	Verify formulation of LCS, re-prepare and re-analyze batch.
Matrix Duplicate	DP	Every 20 samples.	RPD ≤ 20%	The DP is used to assess the effect of the sample matrix on the precision of the method. Evaluate any DP outside limits and note as a non-conformance if a matrix effect is indicated.

Appendix A: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (DP): duplicate aliquot of a sample processed and a Sample Duplicate.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Nonconformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

STANDARD OPERATING PROCEDURE
STL BURLINGTON

THE DETERMINATION OF TOTAL SUSPENDED SOLIDS (TSS) METHOD 160.2

Applicable Matrix: Nonpotable Water

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the determination of Total Suspended Solids (TSS) also known as Non-Filterable Residue in nonpotable water.

1.2 The reporting limit is 0.50 mg/L based on a 1000 mL sample.

2.0 SUMMARY OF METHOD

2.1 The sample is filtered through a pre-weighed standard glass fiber filter disk. The filter disk with residue is placed in an oven maintained at 105^{o}C (±2) and allowed to dry to a constant weight. The concentration of suspended solids is determined by the difference of the initial and final weight measurements of the glass fiber filter disk.

2.2 This procedure is based on Method 160.2, "Residue, Non-Filterable (Gravimetric, Dried at 103-105°C)", Methods for Chemical Analysis of Water and Wastes (MCAWW) - EPA/600/4-79-020 - Revised March 1983.

The laboratory procedure is modified from the reference method as follows:

- The laboratory uses drying oven criteria of 105^{o}C (±2) for this test method.
- The laboratory routinely filters 500-1000mL of sample to ensure collection of at least 1.0 mg of residue.

3.0 DEFINITIONS

Definitions are included in Appendix A.

4.0 INTERFERENCES

4.1 Interferences may be caused by contaminants in the reagent water, reagents, and glassware. To minimize interferences, high purity reagents must be used and all glassware prepared following the procedures described in this SOP.

4.2 Samples high in dissolved solids may be subject to a positive interference.

4.3 Residues dried at 105^{o}C (±2) may retain not only crystallized water, but also some water that is trapped by dried residues, so attainment of constant weight may be very slow. To help eliminate this interference, the sample is dried for a minimum of 16 hours.

5.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.1 Specific Safety Requirements
None

5.2 Primary Materials Used

There are no materials used in this method that have a significant or serious hazard rating **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found Section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 EQUIPMENT AND SUPPLIES

6.1 Glass Fiber Filter Discs without Organic Binder, Whatman 934-AH or equivalent.

6.2 *Note: Because of the physical nature of glass fiber filters, the absolute pore size cannot be controlled or measured. Terms such as "pore size", "collection efficiencies," and "effective retention," are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above.*

6.3 Filtering apparatus equipped with reservoir and a coarse (40-60 microns) fritted disc, Millipore or equivalent.

6.4 Suction Flask, 2000 mL.

6.5 Drying Oven, 105^{o}C (±2).

6.6 Desiccator, Desiccant: Change the desiccant when the "pebbles" have changed color from blue to pink.

6.7 Analytical Balance, capable of weighing of weight measurements to 0.1 mg

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent Water

Infusoil Earth; reagent grade; Fisher Scientific or equivalent

7.2 Standards

LCS (500 mg/L): Measure 1.000g of Infusoil Earth that has been dried at 105^{o}C (±2) for 2 hours, and sieved through #200 (75 μm) mesh sieve into a 2000 mL flask. Adjust to volume with reagent water. Assign an expiration date of 6 months form date of prepared and store at room temperature.

8.0 SAMPLE HANDLING AND PRESERVATION

A minimum sample volume of 100 mL should be collected in plastic or glass unpreserved containers. Immediately following collection all samples should be cooled to 4˚C (±2) and maintained at that temperature until sample preparation and analysis. If Total Dissolved Solids (TDS) by EPA Method 160.1 is also being determined from the same sample, the filtrate generated from this procedure may be used for that procedure.

8.1 The holding time is 7 days from the date of collection.

8.2 Unless otherwise specified by client or regulatory program, samples are retained for 30 days from date of invoice and then disposed of in accordance with Federal, State, and Local regulations.

9.0 QUALITY CONTROL

9.1 A method blank (MB) and a laboratory control sample (LCS) are prepared with each batch of 20 or less samples. A matrix duplicate (DP) is also prepared with each batch of 20 or less samples when sufficient sample volume is available. The acceptance criteria for QC samples are provided in Table 1, Section 18.0 along with recommended corrective action.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Check the calibration of the analytical balance daily prior to use.

11.0 PROCEDURE

11.1 Filter Preparation

Place the glass fiber filter on the membrane filter apparatus with the wrinkled surface facing up. Attach the filter apparatus to the 2000 mL suction flask. Attach the suction flask to the vacuum by securing the vacuum hose to the side arm of the flask. Apply the vacuum at constant pressure and rinse the filter with three successive 20 mL volumes of reagent water. Discard the rinsate and then remove the filter disk from the apparatus and place in an oven maintained at a temperature of 105^{o}C (±2). Keep the filter disks in the oven for one hour. Remove the filter disks from the oven and store until use at ambient temperature in a desiccator.

Tare the calibrated balance and weigh each filter to the nearest 0.1 mg. Record the weight measurement as Filter WT 1 on the bench sheet. Re-weigh the filter after 15 minutes and record the weight as Filter WT 2 on the benchsheet. The difference between the two weight measurements should be $\leq$ 0.5 mg (0.0005 g). If it is not, repeat the drying cycle until the appropriate weight difference is obtained. Handle the filters with forceps or tongs only.

11.2 Sample Preparation and Analysis

Assemble the filtering apparatus and attach the filter apparatus to the 2000 mL suction flask. Attach the suction flask to the vacuum by securing the vacuum hose to the side

arm of the flask. Apply the vacuum at constant pressure. Place a filter onto the filter disk and wet the filter with a small volume of reagent water to secure the filter against the fritted support.

Shake the sample to mix and measure 100 mL into a graduated cylinder. For the method blank, use 100 mL of reagent water. For the LCS use 50 mL of the 500 mg/L Infusoil Earth solution plus 50 mL reagent water. For the matrix duplicate, use a second 100 mL volume of the parent field sample.

Pour the sample aliquot through the funnel of the filtering apparatus using 100 mL aliquot up to 1000 mL of sample until at least 1.0 mg of residue is captured on the filter. It may be necessary to filter less than 100 mL of sample depending on the sample. Record the volume of sample used on the bench sheet. Continue to apply vacuum pressure until all trace of water has passed through the filter. Rinse the graduated cylinder, filter, the non-filterable residue and the filter funnel wall with three successive rinses of reagent water and allow the water to drain completely between each rinse.

Note: If total dissolved solids is also being determined, the filtrate from this step may be used as the sample aliquot for that test.

Remove the filter from the filter support apparatus using tongs or forceps and place in a pre-weighed labeled aluminum dish.

Dry the filter in an oven maintained at a temperature of 105^{o}C ($\pm$2) for at least 16 hours. After 16 hours have elapsed, remove the aluminum dishes from the oven and set them in a dessicator to cool to room temperature.

Tare the calibrated balance and weigh each filter (without aluminum dish) to the nearest 0.1 mg. Record the weight on the bench sheet as Final WT 1. Re-weigh each filter after 15 minutes have elapsed and record the weight as Final WT 2. Remember to tare the balance prior to each filter weight measurement. The difference between the two weight measurements should be $\leq$ 0.5 mg (0.0005 g). If it is not, repeat the drying cycle until a constant weight is obtained.

Calculate the concentration of TSS using the equation given in Section 12.0.

12.0 CALCULATIONS

12.1 Non-Filtered Residue (TSS) as follows:

$$mg/L = \frac{(A-B) * 1{,}000{,}000}{C}$$

Where:
A = Final WT 2 in g
B = Filter WT 2 in g
C = Volume of Sample Filtered in mL

12.2 LCS/MS Percent Recovery (%R)

$$\%R = \frac{SR}{SA} * 100\%$$

Where:
SR= LCS Result
SA= LCS True Value

12.3 Precision (RPD)

$$\%RPD = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} * 100$$

Where:
D1 = Sample result
D2 = Matrix duplicate result

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Review the samples, standards and QC samples against the performance criteria given in Table 1, Section 18.0. If the results do not fall within the established limits perform the recommended corrective action (See Table 1). If corrective action is not taken or unsuccessful, record the situation with a nonconformance report and report and qualify the data as necessary.

14.0 METHOD PERFORMANCE

14.1 Not applicable.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.2 The following waste streams are produced when this method is carried out.

- Aluminum weigh tins and filter residue – Satellite Container: 2.5 L Glass General waste stream

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REVISION HISTORY

16.1 Title page: Updated to reflect current laboratory management team.

16.2 Section 2.0: Added section 2.3.

16.3 Section 7.0: Added Infusoil earth as a reagent material

16.4 Section 9.2: Removed this section.

16.5 Section 11.2: removed reference to method 160.4 (VTS)

16.6 Section 15.2: removed acidic waste from waste stream information

16.7 Section 16.0: Renamed Revision History.

16.8 Section 17.0: Renamed References.

16.9 Section 18.0: Section was added.

16.10 Entire document: changed drying oven criteria to 105^{o}C ($\pm$2).

17.0 REFERENCES

17.1 Method 160.2, “Residue, Non-Filterable (Gravimetric, Dried at 103-105°C)”, Methods for Chemical Analysis of Water and Wastes (MCAWW) - EPA/600/4-79-020 - Revised March 1983.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table1: QC Summary and Recommended Corrective Action
18.2 Appendix A: Terms and Definitions

Table 1: QC Summary and Recommended Corrective Action

QC Item	Acronym	Frequency	Acceptance Criteria	Corrective Action
Method Blank	MB	Each batch of 20 samples or less.	< RL < ½ RL (DoD only)	Re-analyze, if still above re-prepare and reanalyze batch.
Laboratory Control Sample	LCS	Each batch of 20 samples or less.	85-115%	Verify Formulation of LCS, re-prepare and re-analyze batch.
Matrix Duplicate	DP	Every 20 samples.	RPD $\leq$ 20%	The DP is used to assess the effect of the sample matrix on the precision of the method. Evaluate any DP outside limits and note as a non-conformance if a matrix effect is indicated.

Appendix A: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (DP): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

STANDARD OPERATING PROCEDURE
STL BURLINGTON

EPA 415.1 / SW-846 9060, TOTAL ORGANIC CARBON (COMBUSTION)
Applicable Matrix: Non-Potable Water

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the determination of total organic carbon in non-potable water. This SOP is not applicable to TOC in solids.

1.2 The reporting limit (RL) is 1.0 mg/L based on an injection volume of 50 uL.

2.0 SUMMARY OF METHOD

2.1 Organic carbon in sample is converted to carbon dioxide (CO_2) by catalytic combustion and measured directly by an infrared detector. The amount of CO_2 present is directly proportional to the concentration of carbonaceous material in the sample.

2.2 This SOP is based on EPA Method 415.1, Organic Carbon, Total (Combustion or Oxidation), Editorial Revision 1974 and SW846 Method 9060, Total Organic Carbon, Revision 0, September 1986.

The following modification was made to the above reference methods:

- The laboratory does not perform blank subtraction.

3.0 DEFINITIONS

3.1 Definitions are included in Appendix A.

4.0 INTERFERENCES

4.1 Carbonate and bicarbonate represent interferences for this test, and are removed by addition of acid and purging with ultra pure air. This step can result in the loss of volatile organic substances.

4.2 This procedure is applicable to homogeneous samples that can be injected into the combustion column by means of a microliter type syringe. The opening of the syringe limits the maximum size of particle that may be included in the sample.

5.0 SAFETY

5.1 Employees must be trained on and they must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 Specific Safety Concerns or Requirements

The auto sampler has a probe that is sharp; use caution not to stick yourself.

The furnace is very hot and can cause severe burns if touched.

5.3 Primary Materials Used

Table 1 in Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 Total Organic Carbon Analyzer/ Autosampler, Shimadzu TOC 5000A or equivalent.

6.2 Glass Vials, 40 mL, 6 mL

6.3 Auto pipettes

6.4 Volumetric Flasks, Class A, assorted sizes

6.5 Ultra "zero" grade air (compressed cylinder)

6.6 Parafilm

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent Water

Hydrochloric acid, Concentrated: J.T. Baker or equivalent

Hydrochloric Acid Solution (HCl), 2N: Add 42 mL of concentrated HCl to a 200 mL volumetric flask containing approximately 100 mL of reagent water and dilute to volume.

7.2 Standards

Potassium Hydrogen Phthalate: Reagent Grade, J.T. Baker or equivalent

Potassium Hydrogen Phthalate Stock Solution, 1000 mg/L: Measure 2.128 g of potassium hydrogen phthalate into a 1 L volumetric flask and adjust to volume with reagent water. Assign an expiration date of 6 months and store at a temperature of 4°C (±2). Use this solution for the ICAL.

Second Source Standard (ICV/LCS): Second source standard purchased commercially. Dilute the reference standard with reagent water to a concentration near the midrange calibration standard. Record the True Value of the ICV on the bench sheet.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 A minimum sample volume of 40 mL should be collected in glass vials with Teflon caps. Immediately following collection the samples should be preserved to a pH of <2 with sulfuric acid, cooled to 4°C (±2) and maintained at that temperature until time of analysis.

8.2 The holding time for samples is 28 days from the time of collection.

8.3 Unless otherwise specified by client or regulatory program, after analysis, samples are held for a minimum of 30 days and then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 A method blank is analyzed with each sample batch. A second source standard (ICV) is analyzed every 15 samples and a sample duplicate (DP) is analyzed every 10 samples. For this procedure the ICV serves as the laboratory control sample (LCS). Matrix spikes and sample duplicates are performed at a frequency of one pair per twenty samples when sufficient sample volume is provided.

9.2 Analytical results that exceed the calibration range are diluted and reanalyzed. The diluted analysis should result in a determination within the calibration curve, preferably in the top half.

9.3 The acceptance criteria along with recommended corrective action are provided in Table 2, Section 18.0.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibration

Prepare a standard curve initially (during method set-up), after major instrument maintenance such as changing of the column catalyst and when failure of the ICV/LCS indicates re-calibration is necessary. Prepare a series of calibration standards by diluting a known volume of the primary source potassium hydrogen phthalate (KHP) standard in reagent water. The recommended formulation for each calibration standard is provided in the following table:

Standard Curve

Level	KHP Standard Volume Used (mL)	Reagent Water Volume Used (mL)	Final Concentration (mg/L)
Blank	0	100	0
Level 1	0.1	99.9	1.0
Level 2	0.3	99.7	3.0
Level 3	0.5	99.5	5.0
Level 4	1.0	99.0	10.0
Level 5	1.5	98.5	15.0

Turn TOC analyzer on and allow it to warm up before use. Select the TOC button at the bottom of the screen to review the instrument details (operation parameter status). When the combustion column has reached sufficient temperature, an "OK" will be displayed next to the furnace temperature.

Fill the 40 mL vials in the center of the autosampler tray with the calibration standards. Fill the first vial with reagent water, and each subsequent vial with one of the calibration standards such that:

Vial 1 = Blank
Vial 2 = Level 1
Vial 3 = Level 2
Vial 4 = Level 3
Vial 5 = Level 4
Vial 6 = Level 5

Add 20 drops of 2N hydrochloric acid to each calibration standard.

Select "File" then "NEW" to initiate a new sample table. Highlight the first row of the table and select "EDIT", "INSERT STANDARD", then select "NEW". After this selection, a worksheet with three tabs will appear: "Conditions", "Curve" and "Data".

Select the "Conditions" tab and enter the following information

Title:	Date of Calibration (Example 120103.cal)
Filename:	"Date".toc (Example 120103.toc)
Method:	Linear Regression without zero shift*
Analysis Type:	NPOC
Range:	1
Sparge Time:	3
Injection Volume:	50 uL

Select the "Data" tab and in the column entitled "Sample Name" enter the name of each standard as STD1-STD6 in rows 1-6, respectively. In the "Sample ID" column, enter the concentration of the standard as it corresponds with sample name as S0 (Blank), S1 (Level 1), S3, S5, S10 and S15. In the "Vial Location" column, enter S1-S6 (Blank-Level

6) in the row that corresponds to the vial positions along with the concentration of those standards. Select "OK" to return to the filename screen. Enter the filename associated with the calibration and select "OK" to save. Enter the number of injections and maximum number of injections as "4".

Select "Start" to begin analysis. After analysis, print and review the calibration curve. The correlation coefficient must be $\geq$ 0.995 for the calibration to be considered acceptable. If this criterion is not met, the calibration procedure must be repeated prior to further analysis. If the calibration is acceptable, establish the new calibration curve as the basis for quantitation. Select "File" and "New". Highlight the first row and select "Edit", "Insert Sample". Highlight "ICV.met" and select "Open". Select the "NPOC" tab and overwrite the filename of the previous calibration curve with the filename of the new calibration curve. Select "OK" then repeat this step for each standard, blank and sample file.

Place the original hardcopy of the calibration curve in the logbook designated for this purpose and provide a photocopy of the calibration to the inorganic data review group.

11.0 PROCEDURE

11.1 Sample Preparation

Allow the samples to warm to ambient temperature then transfer ~6 mL of each sample into a glass sample tube. Measure additional aliquots for any sample selected as the matrix spike or sample duplicate.

To prepare the matrix spike, combine 0.1 mL of the 1000 mg/L calibration stock standard with 20.0 mL of sample in an empty 40 mL sample vial. True value = 5.0 mg/L.

To prepare the method blank, transfer ~6 mL of reagent water into a glass sample tube. Prepare additional aliquots for every 20 samples.

Transfer ~6 mL of the second source standard to serve as the ICV/LCS. Prepare additional aliquots for every 15 samples.

If analysis of a low-level standard was requested, transfer ~6 mL of the 2.0 mg/L (Level 1) working calibration standard to a sample tube.

Place the vials in the autosampler tray beginning with position 1.

An example sequence analytical sequence is:

ICV/LCS
LLS (per client request)
MB
15 Samples
ICV/LCS

MB
15 Samples
ICV/LCS
MB

Add 3 drops of 2N hydrochloric acid to each sample tube and cover with parafilm to prevent bubbling over during sparging.

Open a new sample table by selecting "File" and "New". Highlight Row #1 and select "Edit" and "Insert Sample". Select the sample type (ICV/LCS, MB, LLS, Sample) that is appropriate to the sample in position 1 and select "OK". Repeat for each blank, standard, sample. In the sample name column, enter the corresponding sample Ids for each field sample. Enter any applicable dilution factor in the Dilution Column.

Save the analytical sequence template with the filename "date.toc" (Example 120103.toc). Place the autosampler cover over the sample tray and select "Start" to initiate analysis. Dilute any samples determined to be above the calibration range, as appropriate.

Analyze all samples in quadruplicate and calculate the average result. Dilute and re-measure samples with concentrations that exceed the range of calibration.

Report the average result from the quadruplicate analysis to the reporting limit (RL) adjusted for sample matrix and sample dilution. The reporting limit is the threshold value below which results are reported as non-detected. Report sample results whose concentration for a target analyte is less than the RL as RL "U".

12.0 CALCULATIONS

Results provided by the instrument software system are in mg/L and corrected for any dilution factors entered into the sample table. The reported result is an average of the four replicates.

12.1 Percent Recovery (%R)

$$\frac{C_s}{C_n} \times 100$$

Where:
C_s = Concentration of the Spiked Sample
C_n = Nominal Concentration of Spike Added

12.2 Percent Recovery (%R) for MS/MSD

$$\frac{C_s - C_u}{C_n} \times 100$$

Where:
C_s = Concentration of the Spiked Sample
C_u = Concentration of the Unspiked Sample
C_n = Nominal Concentration of Spike Added

12.3 Relative Percent Difference (%RPD)

$$\frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where:
C_1 = Measured Concentration of First Sample
C_2 = Measured Concentration of Second Sample

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Review the samples, standards and QC samples against the acceptance criteria given in Table 2, Section 18.0. If the results do not fall within the established limits or criteria, perform the recommended corrective action. If corrective action is not taken or unsuccessful, the situation should be documented and reported in the project narrative. For additional guidance regarding the laboratory's protocol and required elements for each level of data review (primary, secondary, and tertiary) refer to laboratory SOP LP-LB-003 *Data Review*.

14.0 METHOD PERFORMANCE

14.1 A demonstration of analyst capability (IDOC) is required prior to use of this SOP and any time there is a significant change in instrument type, personnel or test method. IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

14.2 A Method Detection Limit (MDL) Study is performed at initial method set-up or when there are significant changes made to the procedure. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-QA-005 *Method Detection Limits.*

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste stream is generated by this method:

- Acidic waste – 10L plastic carboy satellite container

Transfer the waste stream to the satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in laboratory SOP LP-LB-001 *Hazardous Waste*.

16.0 REVISION HISTORY

16.1 Title Page: Changed to reflect current management team.

16.2 Section 18.0, Table 2: Changed ICV/LCS & MS limits from 90-110% to 85-115%

17.0 REFERENCES

17.1 EPA Method 415.1, Organic Carbon, Total (Combustion or Oxidation), Editorial Revision 1974.

17.2 SW846 Method 9060, Total Organic Carbon, Revision 0, September 1986.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Primary Materials Used
18.2 Table 2: QC Summary
18.3 Appendix A: Terms and Definitions

Table 1:Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydradator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 2: QC Summary

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Standard Curve (ICAL)	Initially and when ICV fails	r ≥ 0.995	Correct problem, repeat analysis. If necessary, re-prep standards.
ICV/LCS	ICV: After each ICAL, every 15 samples	%R (85-115)	Correct problem and verify second source standard. If that fails, repeat the standard curve.
Method Blank	Every 15 samples after each ICV/LCS	Target <RL DoD: < ½ RL	Re-analyze, if still above re-prepare and reanalyze batch.
Matrix Spike	1 per 20 samples	%R (85-115)	Examine project DQO's with PM to determine appropriate corrective action; qualify data
Sample Duplicate	1 per 20 samples	RPD ≤ 20	Examine project DQO's with PM to determine appropriate corrective action; qualify data
Low Level Standard	Per client request	Per client specification	Per client specification

Appendix A: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): solution prepared from a separate source from that which is used to prepare the calibration curve.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (DP): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

STL BURLINGTON
STANDARD OPERATING PROCEDURE

TOTAL ORGANIC CARBON IN SOILS AND SEDIMENT
Lloyd-Kahn Method
Applicable Matrix: Soils, Sediments, and Other Solids

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the determination of total organic carbon (TOC) in soils, sediments and other solids. A procedure for the determination of TOC in marine sediment high in inorganic carbon is provided in Appendix B.

1.2 The routine reporting limit is 500 mg/kg. Additional sample may be used (up to 25 mg) to achieve as low a reporting limit as 100 mg/kg.

2.0 SUMMARY OF METHOD

2.1 A small aliquot of sample, routinely 10.0 mg, is transferred to a tin capsule and treated with phosphoric acid, then dried in an oven at 105°C for 30 minutes to one hour. This serves to separate the organic carbon from inorganic carbonates and bicarbonates. The sample is then transferred to an instrument where it is pyrolyzed in an inductive type furnace. The carbon is converted to carbon dioxide and measured by a differential thermal conductivity detector.

2.2 This procedure is based on the EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1998, authored by Lloyd Kahn, Quality Assurance Specialist.

3.0 DEFINITIONS

3.1 Definitions are included in Appendix A.

4.0 INTERFERENCES

4.1 Volatile organics in the sediments may be lost in the decarbonation step resulting in a low bias. Maintaining the sample at 4°C, analyzing the sample within the specified holding time, and analyzing the wet sample, may minimize bacterial decomposition and volatilization of the organic compounds.

5.0 SAFETY

Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.1 Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1, Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary

hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

Drying Oven: Capable of maintaining a temperature of 105°C.

Carlo Erba Elemental Analyzer Model EA1108 and Model NA 1500 or equivalent.

Costech Elemental Analyzer: Model 4010 or equivalent.

Analytical Balance: Capable of weighing to the nearest 0.001mg.

Aluminum Trays that hold sample capsules for use at 105°C

Tweezers

5mm X 9mm tin capsules

Quartz Columns: Costech Analytical or equivalent.

Quartz wool: for segregating and containing column materials

Copper Wire, Reduced: Costech Analytical or equivalent.

Tungsten on Alumina: Costech Analytical or equivalent.

High Temperature Gloves

Clear Plastic Sample Trays: Costech Analytical or equivalent.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Reagent water

Phosphoric Acid, Concentrated: Reagent Grade, J.T. Baker recommended.

Phosphoric Acid Solution (1:19): Add approximately 100 mL of reagent water to a 200 mL volumetric flask. Add 18.34 g of concentrated phosphoric acid to the volumetric flask then adjust to volume with reagent water. Mix the solution well then transfer the solution to a 250 mL polyethylene bottle. Assign an expiration

date of six months from date made and store the solution at room temperature.

7.2 Standards

Acetanilide Crystals of known Carbon percentage: Purchased from Costech Analytical. Used to check instrument calibration.

Sulfanilamide Crystals (41.84% Carbon): Purchased from Costech Analytical. This material is used to calibrate the instruments.

Laboratory Control Samples (LCS) Material, Organic Material of known Carbon percentage: Purchased from LECO Corporation.

Matrix Spike Material, 1632B trace elements in coal (80.11% Carbon)

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT & STORAGE

8.1 Samples should be collected in amber glass jars. Immediately following collection, the samples should be cooled to 4°C (±2) and maintained at that temperature until time of analysis.

8.2 The holding time is 14 days from date of collection, unless otherwise specified.

8.3 Unless otherwise specified by a federal, state or client-specific protocol, samples are disposed of after 30 days in a manner that complies with all applicable regulations.

9.0 QUALITY CONTROL

9.1 The following QC check samples are analyzed with each batch of 20 or less samples: Method Blank (MB) Laboratory Control Sample (LCS), Matrix Spike (MS) and a Sample Duplicate (DP). In addition to calibration (ICAL), instrument standardization is checked with acetanilide every 20 drops and at the end of the analytical sequence. The minimum frequency requirements, acceptance criteria and recommended corrective action for QC samples are summarized in Table 2, Section 18.0.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibration Curve

Analyze a calibration curve each time the combustion column is changed (a combustion column is good for about 200 sample drops). The recommended formulations for each calibration level is provided in the following table:

Calibration Standard Sulfanimide	Weight[1] (mg)	% Carbon	Carbon (mg)
Calibration Level 1	0.100	41.84	0.0418
Calibration Level 2	0.500	41.84	0.2092
Calibration Level 3	1.00	41.84	0.4184
Calibration Level 4	1.75	41.84	0.7322
Calibration Level 5	2.50	41.84	1.046

[1] *These weights are approximate. Enter the actual weight used into the software program.*

Measure a single drop for each calibration point. The instrument software system plots peak area against mg of Carbon and calculates a correlation coefficient using standard linear regression. The correlation coefficient (r) must be $\geq$ 0.995 for the calibration to be considered acceptable. If it is not, repeat the calibration prior to further analysis.

11.0 PROCEDURE

11.1 Sample Preparation

Using tweezers, and working directly from the box, place a tin capsule on the analytical balance and tare. Using the small sample scoop, add approximately 10 mg (or more, if client requested) of sample to the capsule. Record the sample weight on the benchsheet. Remove the capsule from the balance and place into one of the aluminum holding trays. Weigh three separate aliquots into three separate tin capsules for each field sample. Record all weight measurements on the sample preparation log. For the method blank, set three empty tin capsules into an aluminum holding tray. For the LCS, weigh ~9 mg of LECO LCS material into four separate tin capsules and set them in sequence in an aluminum holding tray.

For the matrix spike, weigh out an additional sample aliquot and record its weight. Add 0.3 – 0.7 mg of matrix spike material and record this weight. For the sample duplicate, weigh out an additional sample aliquot. Prepare three aliquots for both the matrix spike and the sample duplicate.

Add two drops of 1:19 phosphoric acid to each tin capsule. Place the aluminum trays into a drying oven set to a temperature of 105ºC for 30-60 minutes or until all samples appear dry.

Using tweezers, pinch the top of each tin capsule closed and compress the capsule around the material inside. Work carefully so as not to tear the capsule, but crush it down to the smallest size. Set the prepared samples in line in a clear plastic sample tray for storage, or place directly into an autosampler tray for analysis. For the latter, leave positions open for the acetanilide check standards and associated calibration blanks.

Prepare the acetanilide standard and blanks as follows:

For each acetanilide spike, weigh ~0.5 mg of acetanilide material into a tin capsule. Fold the capsule up and compress down to the smallest size possible. Prepare enough acetanilde to ensure a frequency of every 20 drops and the end of the analytical sequence. For each associated calibration blank, leave an empty position in the autosampler tray.

11.2 Software Set-up and Analysis

If the column has been changed, generate a new calibration curve. If not, use the existing calibration curve for analysis. Each column will analyze approximately 200 individual sample drops. When the counter on the instrument approaches 200, watch the instrument data for signs that the column is deteriorating; poor peak resolution, trailing baselines, extraneous peaks. If a column change is necessary, refer to Appendix C for the procedure. After changing the column, generate a new calibration curve.

Select the appropriate channel: Channel 1 is the NA 1500, Channel 2 is the EA 1108, and Channel 3 is the Costech instrument, which has its own PC. At the main screen select the sample table icon. The last sample table that was run will be shown on the screen.

Open a new sample table, and select the appropriate number of sample positions for the analysis, then name the table with the date and a unique alpha designator (i.e. 061505a). In front of the %3r in the file name column of the sample table, add the sample table name to ensure that each individual chromatogram generated from this sample table has a unique filename associated with it.

If the combustion column has been changed and instrument needs to be calibrated, follow the procedure below:

Prepare a “bypass” drop to determine the retention time for carbon with the new column. The bypass is an aliquot of acetanilide. The weight is not needed. Drop the bypass into the instrument and initiate a singular analysis. Set the retention time for carbon in the software to match that of the bypass drop.

Identify the first five sample lines with the names Std1 through Std 5. Enter their respective weights in the weight column, assign them a level # in the level column (Std1 is level 1, Std2 is level 2, etc.) to alert the software the order in which to place the calibration standards. In the sample type column, use the drop down and select “standard” for each. Finally, use the drop down in the Standard name column and select “sulfanilamide” for each. Add the standards to the autosampler tray and hit “start” to run the calibration.

Sample Analysis:

Open a new sample tray and create a unique file name. When the instrument was last calibrated, the software create a calibration file with the same name as the sample table in which it was run. Open this file and save it with the same name as the sample table about to be run to ensure that the analysis is calculated from the most recent calibration. To do this, click on the calibration icon (looks like a little calibration curve) and use the file option to open the calibration file last performed. Save this file with the same name as your sample table. Click on the sample table icon (looks like a little sample table) to get back to your sample table.

Enter each sample ID and their respective weights and save the sample table. Enter a weight of 10 mg for the Method Blank (PBS) and instrument blanks.

An example analytical sequence follows:

Initial Calibration (calibration blank and 5 calibration standards)

Acetanilide	(1 drop)
Blank	(1 drop)
PBS	(3 individual drops)
LCS	(4 individual drops)
Sample	(3 individual drops)
Sample	(3 individual drops)
Sample	(3 individual drops)
Sample	(3 individual drops)
Sample	(1 individual drop)
Acetanilide	(1 drop)
Blank	(1 drop)

Add the samples and acetanilides to the autosampler tray and set the tray into the autosampler carriage. Turn the autosampler tray until the number 1 position is behind the post, in front of the autosampler. The tray is now set to run.

Click the “start” icon to begin the analysis

After analysis review the analytical results against the acceptance criteria given in Table 2, Section 18.0, and perform corrective action as necessary. Enter the results for all instrument blanks (including PBS) and any client sample exhibiting an area response at or lower than the lowest calibration standard into the low level Excel spreadsheet set up for this purpose. This spreadsheet calculates these low level results with a two point linear regression using the origin and the lowest calibration point. A more precise result for low level samples is determined this way. Report results in mg/kg Carbon and corrected for % solids

12.0 CALCULATIONS

12.1 Percent Carbon to mg/kg Carbon Conversion

% Carbon × 10,000 = mg/kg Carbon

12.2 LCS Percent Recovery (%R)

$$\%R = \frac{\text{LCS Result}}{\text{LCS True Value}} \times 100$$

12.3 MS Percent Recovery (%R)

$$\text{mg/Kg wet SA} = \frac{\text{Spike TV} \times \text{weight of MS added}}{\text{sample weight}} \times 1\text{million}$$

$$\text{mg/Kg dry SA} = \frac{\text{mg/Kg wet SA}}{\%\,\text{solid}} \times 100$$

$$\text{mg/Kg dry Carbon} = \frac{\text{mg/Kg wet Carbon (from instrument)}}{\%\,\text{solid}} \times 100$$

$$\%R = \frac{A - B}{C} \times 100$$

Where:
A= Average of three drops of MS sample result: mg/Kg dry carbon
B= Average of three drops of parent sample: mg/Kg dry carbon
C= Average of three drops of mg/Kg dry SA
SA= spike added (mg/Kg)
Spike TV= 0.8011(mg/Kg)

12.4 Relative Percent Difference (RPD)

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

Where:

D_1 = First Sample Value

D_2 = Second Sample Value (duplicate)

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Review the samples, standards and QC samples against the performance criteria given in Table 2. If the results do not fall within the established limits or criteria perform corrective action. If corrective action is not taken or unsuccessful, the

situation should be documented and reported in the project narrative. All data that does not meet established criteria must be flagged and noted in the project narrative.

14.0 METHOD PERFORMANCE

14.1 An Initial Demonstration of Capability is required for each analyst before unsupervised performance of this method.

14.2 An Initial Method Detection Limit (MDL) determination for each test method referenced in this SOP is performed following the procedure described in the reference method, 40CFR, Part 136, Appendix B and laboratory SOP LP-LB-009. The MDL is verified or repeated when a significant change to the method occurs. Significant changes include the use of alternate reagents or standard reference materials, new instrumentation or the use of alternate sample preparation procedures.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 The laboratory optimizes technology to minimize pollution and reduce the production of hazardous waste whenever possible.

15.2 Waste Streams generated by this method;

- Spent combustion columns

→ *Satellite Waste Container: Five Gallon Metal Bucket labeled "Glass Disposal"*

Transfer the waste stream to the appropriate hazardous waste satellite container located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite container to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in laboratory SOP LP-LB-001 Hazardous Waste.

16.0 REVISION HISTROY

16.1 Cover Page: Changed to reflect current management team.
16.2 Section 2.1: changed routine sample mass from 5.0 mg to 10.0 mg
16.3 Section 6.0: added Costech analyzer
16.4 Section 7.2 & 11.1: added matrix spike material and prep procedure
16.5 Section 11.1: hydrochloric acid reference changed to phosphoric
16.6 Section 18.0, Table 2: changed acetanilide criteria to 85-115%

17.0 REFERENCES

17.1 EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1998, authored by Lloyd Kahn, Quality Assurance Specialist.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Primary Materials Used
18.2 Table 2: QC Summary
18.3 Appendix A: Definitions
18.4 Appendix B: TOC Procedure for High Concentration Marine Sediments (CITHON)
18.5 Appendix C: Column change procedure

Table 1: Primary Materials used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			

Table 2: QC Summary and Recommended Corrective Action

QC Sample	Frequency	Acceptance Criteria	Corrective Action
ICAL	Following each column change	$r \geq 0.995$	Standards check, re-calibration.
Method Blank (PBS)	Once per batch of 20 samples	< RL	Re-prepare and reanalyze batch.
LCS	Once per batch of 20 samples	%R (75-125)	Re-prepare and reanalyze batch.
Acetanilide	Every 20 drops and at the end of the analytical run	%R (85-115)	Re-prepare and reanalyze samples not surrounded by passing Acetanilides
Blank (paired with Acetanilide)	Following each Acetanilide	< RL	Re-prepare and reanalyze batch.
Matrix Spike	One per batch of 20 or less samples	%R (75-125)	Discuss outlier in project narrative
Sample duplicate	One per batch of 20 or less samples	RPD < 20	Discuss outlier in project narrative

Appendix A: Definitions

Accuracy: the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria.

Calibration: the establishment of an analytical curve based on the absorbance, emission intensity or other measured characteristic of known standard.

Calibration Blank (ICB/CCB): a volume of reagent water acidified with the same acid matrix as in the calibration standards.

Calibration Standards: a series of known standard solutions used to calibrate the instrument response with respect to analyte concentration. A standard containing the analyte in question (sulphanilimide) is prepared at varying weights and analyzed. This standard is a separate source from the LCS. The sulphanilimide is used to calibrate the instrument response with respect to analyte concentration.

Continuing Calibration Verification (CCV): a prepared standard solution used to verify the stability of the instrument calibration and instrument performance during the analysis of samples.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): A prepared standard solution from a source separate from that of the calibration standards used to verify the concentration of the calibration standards and the adequacy of instrument calibration.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as samples, through all steps of the analytical procedure.

Matrix: the substrate of a test sample.

Matrix Duplicate (DP): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is +100%. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

Percent Solids (%S): the proportion of solid in a soil sample.

Appendix B: Marine Sediments High in Inorganic Carbon

Sample Preparation

Transfer approximately 10 g of a thoroughly mixed sample to an aluminum weigh dish, and dry in the 105°C oven. Grind the sample with the pink mortar and pestle to a fine powder. Record the weight of a 250 mL Teflon beaker then transfer ~ 5 g of the ground sample to this beaker.

If the sample is to be spiked, weigh the beaker to the nearest 0.1mg and record the weight. Likewise determine and record the weight of the added sample. Add 0.1g of NIST 1632b Trace Elements in Coal (80.11% Carbon) to the sample. Record the weight added. Evenly distribute the spike over the sample and use a glass stir rod to mix the spike with the sample. Do not use that stir rod with any other sample.

Use Talc-free latex gloves from this point on to minimize the risk of acid burns. Add several drops of 1:1 HCL to each sample and stir each sample with its own glass stir rod. Samples with high concentrations of inorganic carbon may effervesce to the point of overflowing the beaker, so take care to add the acid in small aliquots and stir vigorously. If the sample "boils over" it must be re-prepared. Continue to add 1:1 HCL in small aliquots until there is no further reaction, taking sample to dryness after each addition of acid with the hot plate provided.

Carefully rinse the stir rod and beaker walls with DI water using a fine-tipped squirt bottle.
Use only what is needed to bring the entire sample to the bottom of the beaker. ***When adding water to acid use necessary precautions to avoid splashing!***

Dry the treated samples on the hot plate in the hood, after each acid/water addition. Do not add more than a total of 200 mL of 1:1 HCL to any sample.

NOTE*: Samples are hydroscopic and will absorb water if they are exposed to air for too long.*

Weigh beaker with residue and record the residue weight measurement. After the sample is thoroughly dry, scrape the sample residue from the beaker and grind to a powder using the pink mortar and pestle. Transfer the ground sample to a clean, dry 40-mL vial reserved for this analysis.

NOTE: *Depending on the nature of the sample, it may be difficult to completely remove the dried residue from the beaker or to grind it to a homogenous powder. Where difficulties are encountered, make a note on the preparation worksheet.*

Analysis

Perform TOC analysis on processed sample material as outlined in section 10.0 of this SOP.

Appendix C: Column Change Procedure

Turn off the helium and oxygen supplies to the instrument.

Dial the left furnace temperature to a reading of 052 (this equates to 520˚C). Wait until the temperature drops below 600˚C to remove the column.

Remove the panel covering the furnace and unscrew the autosampler connection from the top of the column.

Unscrew the fitting at the bottom of the column and remove.

Lift the column up and out of the furnace using high temperature gloves.

CAUTION: The column will still be 500-600˚C. Do not touch the center portion of the column. Place the spent column in the metal can designated for this purpose.

Lay a new quartz column on the bench top, measure and mark off for the following:

- One inch up from the bottom and add a ½ inch plug of quartz wool. Note: pack the quartz wool tightly enough for it to stay in place.
- Pour in 2 ½ inches of copper wire
- Pack another ½ inch quartz wool plug on top of the copper
- Pour in 3 inches of tungsten
- Pack a final ½ inch quartz wool plug on top of the tungsten

Place the new column into the furnace and reconnect the top and bottom fittings. Snug these up, but don't over tighten.

Replace the panel covering the furnace, dial the furnace temperature back to 102 (this equates to 1020˚C), and turn the helium and oxygen supplies back on.

When the instrument comes up to operating temperature, it is ready to calibrate.

STANDARD OPERATING PROCEDURE
STL BURLINGTON

ACID VOLATILE SULFIDE & SIMULTAENOUSLY EXTRACTED METALS (AVS/SEM)
Applicable Matrix: Sediment

APPROVAL SIGNATURES

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the determination of acid volatile sulfide (AVS) and for selected metals that are solubilized during the acidification step (simultaneously extracted metal, SEM). The metals for SEM evaluation typically include cadmium, copper, lead, mercury, nickel, and zinc, although this list may be amended on a project basis.

1.2 The reporting limit for AVS is 16mg/Kg based on a 10g sample weight. For trace metals, results are reported to the instrument detection limit (IDL), which are established quarterly. SEM extracts for ICP-AES typically require a 10-fold dilution to accommodate for the difference in acid solutions between SEM extracts and those used for instrument calibration and the IDLs are adjusted accordingly. The reporting limit for SEM extracts for mercury is 0.025mg/Kg based on a 10g sample weight.

2.0 SUMMARY OF METHOD

2.1 The AVS in the sample is converted to hydrogen sulfide by reaction with a 6N hydrochloric acid solution in a closed system and at room temperature. The hydrogen sulfide is purged from the sample and trapped in a sodium hydroxide solution. Quantitation of sulfide is based on the titration of the sodium hydroxide solution using iodine and sodium thiosulfate. Additionally, after the reaction is complete, the liquid phase in the reaction flask is filtered and SEM is determined by ICP-AES analysis and, as required, by CVAA Mercury analysis following the procedures given in laboratory SOPs LM-MI-6010B and LM-HG-7470, respectively.

2.2 This procedure is based on the USEPA Office of Water, Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment, December 1991.

2.3 The following method modifications were made:

2.4 The laboratory uses a determinative method for sulfide that is based on SW846 Method 9034, which is a titrimetric method using iodine and sodium thiosulfate. The referenced method provides for the determination of sulfide by either a gravimetric, a colorimetric, or an ion-selective probe method. The titrimetric method is used in the referenced method as the bases for critically evaluating the calibration standards that are employed in performing any of the three determinative methods.

3.0 DEFINITIONS

3.1 Definitions are included in Appendix A.

4.0 INTERFERENCES

4.1 Samples must be collected with a minimum of aeration to avoid volatilization of sulfide or reaction with oxygen, which oxidizes sulfide to sulfur compounds that are not detected.

This same caution is particularly applicable to the handling of samples in the laboratory.

4.2 Reduced sulfur compounds, such as sulfite and hydrosulfite, decompose in acid, and may form sulfur dioxide. This gas may be carried over to the zinc acetate gas scrubbing bottles and subsequently react with the iodine in the determinative step to yield false high values. This method shows no sensitivity to sulfite or hydrosulfite at concentrations up to 10 mg/kg of the interferent.

4.3 Reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds can interfere with the iodometric-titrimetric method.

5.0 SAFETY

5.1 Employees must be trained on and they must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 Specific Safety Concerns or Requirements

Sodium Sulfide will form Hydrogen Sulfide (HS) gas if combined with water moisture or strong acids. Inhalation of HS gas may be fatal.

5.3 Primary Materials Used

Table 1 provided in Section 18.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 Top Loading Balance, capable of measurement to the nearest 0.01g

6.2 Stir Plate

6.3 Teflon Coated Stir Bar

6.4 Round-Bottom Double-Neck Distillation Flask – 500 mL, 24/40 standard taper joints.

6.5 Funnel with Polytetrafluoroethylene (PTFE) Plug.

6.6 Thistle Tube – 24/40 outlet joint.

6.7 Impingers with Stone Frit (two, aligned in series).

6.8 Tubing – ¼ in. PTFE or polypropylene (do not use rubber).

6.9 Nitrogen flow regulator.

6.10 Wide range pH paper, pH 0-14.

6.11 Volumetric flasks, 50, 100, 500 mL.

6.12 Erlenmeyer flasks, 250 mL.

6.13 Graduated cylinders, 100 mL.

6.14 Burette, 50 mL.

6.15 Volumetric Pipettes, 1.0, 2.0, 5.0, 10.0, 50.0 mL.

6.16 Filters, 0.20 micron: acid washed.

7.0 REAGENTS AND STANDARDS

Unless otherwise noted, assign an expiration date of six months from date of preparation unless the expiration date of the parent component expires sooner, in which case use the earliest expiration date. Store prepared reagent and standard at room temperature unless otherwise noted.

7.1 Reagents

Reagent Water: *deaerated by bubbling with nitrogen for one hour prior to addition into sample flasks*

Nitrogen Gas Source

Nitric Acid, Concentrated: Reagent Grade, J.T. Baker or equivalent

Hydrochloric Acid (HCl), Concentrated: Reagent Grade, J.T. Baker or equivalent

Sulfuric Acid (H_2SO_4), Concentrated: Reagent Grade, J.T. Baker or equivalent

Sodium Hydroxide Pellets: Reagent Grade, J.T. Baker or equivalent

Zinc Acetate ($Zn(CH_3COO)_2 \cdot 2H_2O$): Reagent Grade, J.T. Baker or equivalent

Potassium Iodide: Reagent Grade, J.T. Baker or equivalent

Sodium Thiosulfate Crystals: Reagent Grade, J.T. Baker or equivalent

Starch Solution, 1.0%: Reagent Grade, Fisher Scientific or equivalent

Iodine Solution, 0.1N: Reagent Grade, Fisher Scientific or equivalent

Sodium Thiosulfate Solution, 0.1N: Reagent Grade, Fisher Scientific or equivalent

Potassium bi-Iodate Solution, 0.1N: $KH(IO_3)_2$: Reagent Grade, Fisher Scientific or equivalent.

Nitric Acid (HNO_3), 25%: Add 125 mL of concentrated nitric acid to 375 mL of reagent water.

Sulfuric Acid (H_2SO_4), 50%: Add 250 mL of concentrated sulfuric acid to 250 mL of reagent water.

Hydrochloric Acid (HCl), 6N: Add 500 mL of concentrated HCl into 500mL reagent water. *Deaerate this solution prior to use for one hour by bubbling nitrogen through it.*

Sodium Hydroxide Impinger Solution, 0.5N: Add 40 g of sodium hydroxide pellets to a 2000 mL volumetric flask that contains ~1000 mL of reagent water. Swirl to mix and bring to final volume with reagent water. Assign an expiration date of six months and store at room temperature.

Iodine Solution, 0.025N: Dilute 500 mL of 0.1N Iodine solution in 2 L of reagent water. Alternatively, dissolve 50 g of potassium iodide in 1400 mL of reagent water then add and dissolve 6.4 g Iodine, (I_2) to the potassium iodide. Adjust the volume to 2 L with reagent water. Standardize the prepared solution each day of use following the procedure given in Section 10.0.

Sodium Thiosulfate Tirtrant ($Na_2SO_3 \cdot 5H_2O$), 0.025N: Dilute 500mL of 0.1N sodium thiosulfate solution in 2 L of reagent water. Alternatively Add 1.5 g NaOH and 20.0g ± 0.01 g sodium thiosulfate crystals , to a pre-weighed plastic container and cap. Add 3220g of reagent water that has been boiled and cooled. Mix thoroughly until the reagent is dissolved. Standardize this solution each day of use following the procedure given in Section 10.0.

7.2 Standards

Mercury Standard Solution (LCS/MS), 1000 ug/L: SPEX or Inorganic Ventures

Trace Metals Standard Solution (LCS/MS), 100 mg/L: SPEX or Inorganic Ventures

Silver Standard Solution (LCS/MS), 100mg/L: SPEX or Inorganic Ventures

Sodium Sulfide Crystals (Na_2S): Reagent Grade, J.T. Baker or equivalent

Sodium Sulfide Stock Solution [$Na_2S \cdot 9H_2O$] (LCS/MS): Add approximately 0.5 g of sodium sulfide crystal to a 500 mL volumetric flask and bring to volume with reagent

water. Determine the concentration of this standard each day of use following the procedure given in Section 10.0.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1 A minimum of 60 grams of sample should be collected in a wide mouth glass container so as to minimize headspace. Immediately following collection, all samples should be cooled to 4˚C (±2) and maintained at that temperature until sample preparation and analysis.

8.2 The holding time for the AVS determination is 14 days from the date of collection. Although the method recommends a 2-week timeframe for the analysis of the SEM extracts, the laboratory uses the holding time specifications in the referenced SW846 methods for the analysis of trace metals and mercury.

8.3 Unless otherwise specified by client or regulatory program, after analysis, samples are retained for a minimum of 30 days after date of submission of the data report and then disposed of in accordance with Federal, State, and Local regulations.

9.0 QUALITY CONTROL

9.1 A method blank (MB) and laboratory control sample (LCS) are analyzed with each batch of 20 or less samples. A matrix spike (MS) and a sample duplicate (SD) should also be analyzed with each batch when a sufficient sample is provided. The acceptance criteria along are provided in Table 2, Section 18.0 along with the recommended corrective actions for when criteria are not met.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Standardization of Sodium Thiosulfate Titrant, 0.025 N:

The standardization process using potassium bi-iodate serves as a second source verification of the reagents used in this procedure.

Pipet 5.0 mL of 0.1N potassium bi-iodate solution into a 500 mL Erlenmeyer flask. Add approximately 1 g of potassium iodide crystals and 2 mL 50% H_2SO_4, adjust the volume to 200 mL with reagent water. Swirl to mix, and let the solution stand in the dark for 5 minutes. Using the prepared sodium thiosulfate titrant, titrate the bi-iodate from yellow to straw color. Add approximately 1 mL of starch solution and titrate from blue to clear endpoint. The volume of titrant should approximate 20 mL.

Calculate the normality:

$$\text{N Thiosulfate} = \frac{(0.1\text{N bi - Iodate}) \times 5.0\ \text{mL}}{\text{mL Thiosulfate}}$$

The calculated normality should be 0.025 ± 0.00025N.

If the solution is greater than 0.02525N, add a proportionate amount of reagent water to the volume of solution being adjusted. Use boiled / cooled reagent water. Repeat the titration of the potassium bi-iodate solution. The volume of thiosulfate must be between 19.8 and 20.2 mL. This corresponds to an acceptance range of 0.02475N - 0.02525N. If the calculated result exceeds 0.02525N, repeat the preceding steps. If the result is less that 0.02475N, add 1.0 g thiosulfate crystals, mix to dissolve, and repeat the titration.

10.2 Standardization of Iodine Solution, 0.025N:

Dissolve approximately 2 g potassium iodide crystals in 150 mL of reagent water. Add 20 mL of the prepared 0.025N iodine solution and adjust the volume to 200 mL with reagent water, then add 1 mL of 6N HCl.

Titrate the solution with the standardized 0.025N sodium thiosulfate titrant (using starch indicator, endpoint = blue to clear).

Calculate the normality:

$$\text{Normality } (I_2) = \frac{\text{mL Titrant} \times \text{N Titrant}}{\text{mL Iodine Solution}}$$

10.3 Determination of Concentration of Sulfide LCS/MS Standard Solution:

To an Erlenmeyer flask add 45 mL reagent water, 5 mL of prepared sulfide stock solution, 10 mL of standardized 0.025N iodine solution, and 2 mL of 6N HCl. If all of the iodine is reduced by the sulfide present, add a second 5.0 mL aliquot of iodine solution. If necessary, continue to add the iodine solution in 5.0 mL increments until amber color remains. Record the total volume of iodine solution added. Add starch solution, and titrate against the standardized 0.025N sodium thiosulfate solution.

Calculate the concentration of the standard solution:

$$\text{LCS/ MS Concnetration (mg/L)} = \frac{(\text{mL Iodine Solution - net mL Thiosulfate Solution}) \times 0.025\text{N} \times 16000}{\text{mL Stock Titrated}}$$

11.0 PROCEDURE

11.1 Distillation Apparatus Assembly

Assemble necessary glassware and equipment. Prior to use, rinse the glassware with 25% nitric acid solution and then reagent water in order to reduce the possibility of contamination.

Add 20 mL of 6N HCl to each addition funnel.

Add 50 mL of 0.5N Sodium Hydroxide solution to each impinger. Two impingers are required for each distillation assembly. Label the impingers, using the position of the

distillation assembly and the suffix “A” for the front impinger, and the suffix “B” for the back impinger. For example, the impingers in the third set-up would be labeled “3A” and “3B”. Connect the impingers in series with tubing, making certain that the outlet and inlet ends are correctly aligned.

11.2 Sample Preparation

To prepare the method blank, add 100 mL of de-aerated reagent water to a labeled distillation flask. Add a stir bar and connect the flask to the distillation assembly.

To prepare the LCS for AVS, add 100 mL of de-aerated reagent water to a labeled distillation flask. Add 10.0 mL of the LCS/MS sulfide standard solution to the flask. Add a stir bar and connect the flask to the distillation assembly. *Note: A solid LCS material is not presently available for acid soluble sulfide.*

To prepare the LCS for SEM, add 100 mL of de-aerated reagent water to a labeled distillation flask. Add 2.5 mL of the LCS/MS mercury standard solution, 6.25 mL of the LCS/MS trace metals standard solution, and (if analysis for silver is required) 2.5 mL of the LCS/MS silver standard solution to the flask. Add a stir bar and connect the flask to the distillation assembly.

For each sample, place a labeled distillation flask onto a calibrated top loading balance. Tare the balance and measure 10 ± 0.5g of sample into the flask. Record the sample weight on the bench sheet. Add 100 mL of deaerated reagent water to the flask. Add the stir bar, and connect the flask to the distillation assembly. For an efficient distillation, the mixture in the distillation flask must be of such a consistency that the motion of the stirring bar is sufficient to keep the solids from settling. The mixture should be free of solid objects that could disrupt the stirring bar.

Weigh an additional aliquot of the sample selected for the MS for AVS into a labeled distillation flask. Record the sample weight on the bench sheet. Add 10.0 mL of the LCS/MS sulfide standard solution to the flask and add 100 mL of deaerated reagent water to the flask. Add the stir bar, and connect the flask to the distillation assembly.

Weigh an additional aliquot of the sample selected for the MS for SEM into a labeled distillation flask. Record the sample weight on the bench sheet. Add 2.5 mL of the LCS/MS mercury standard solution, 6.25 mL of the LCS/MS trace metals standard solution, and 2.5 mL of the LCS/MS silver standard solution to the flask. Add 100 mL of deaerated reagent water to the flask. Add the stir bar, and connect the flask to the distillation assembly.

Weigh an additional aliquot of the sample selected for the sample duplicate into a labeled distillation flask. Record the sample weight on the bench sheet. Add 100 mL of reagent water to the flask. Add the stir bar, and connect the flask to the distillation assembly.

11.3 Distillation

Assemble the distillation train and ensure that all connections are tight. All joints, both ground glass and tubing, should be wrapped in parafilm before proceeding. Start the flow of nitrogen and adjust the flow rate through the system to 60 mL/min, and ensure that there is a good flow of gas through the impingers. Purge the system of oxygen for ~15 minutes.

Slowly open the funnel stopcock and allow the 6N HCl to mix with the sample. Watch for possible reactions such as a rapid burst of bubbles, foaming, or bumping. Be prepared to stop or slow the addition of HCl until any reaction has subsided. The HCl transfer should be complete in about two minutes.

Reduce the nitrogen flow to 20 mL/min.

Set the timer for 60 minutes. During this time, frequently check flasks for adequate stirring and check impingers for consistent nitrogen flow.

Following the 60-minute reaction time, turn off heat and nitrogen gas, and disassemble the apparatus. Using wide-range pH paper, check the pH of each distillate, and record the pH value.

11.4 Titration

Titrate the impinger fluid directly in the impinger. Use a pipette bulb to push the any solution contained in the fritted bubbler chamber back through the frit, into the main chamber of the impinger.

Add 5.0 mL of 0.025N standardized iodine solution and 1mL of 6N HCl to the impinger. If all the iodine is reduced by the sulfide present, continue to add 5.0mL aliquots of iodine until the solution changes to the dark red color of iodine. Record the total volume of iodine added on the bench sheet.

Titrate with the standardized 0.025N sodium thiosulfate solution to a straw color. Add approximately 1mL of starch solution and titrate from blue to clear endpoint. Record the initial and final volume of titrant.

Repeat the preceding steps for each impinger, titrating the “front” and “back” positions for each distillation assembly separately.

Calculate the results using the equation given in section 12.0. The back impinger result should be less than or equal to 25% of the front impinger result.

11.5 SEM Sample Preparation

Filter the sediment suspension in the distillation flask through an acid washed 0.20 micron filter. This requires a quantitative transfer of the aqueous phase, which can be accomplished by an initial decanting and three successive 10 mL rinses of the solid

phase with reagent water. Although there is no solid phase associated with the method blank or the LCS, the same filtration and rinse procedure should be followed in preparing these sample volumes. (This step is not to be performed on the LCS or MS volumes specific to the AVS).

Transfer the filtrate to a 250 mL volumetric flask. Rinse the filtering flask with reagent water and add the rinse to the 250 mL volumetric flask, adjust to volume with reagent water.

11.6 SEM Digestion and Analysis

Analyze the sample filtrates using the procedures given in laboratory SOPs for LM-MI-601B for ICP-AES and/or LM-HG-7470 for CVAA Mercury. Calculate the AVS and SEM results, and the SEM:AVS ratio using the equations given in Section 12.0.

12.0 CALCULATIONS

12.1 AVS Calculation:

12.2

$$\text{mg/Kg } S^{2-} \text{ (Front Impinger)} = \frac{(\text{mL } I_2 \times \text{N } I_2) - (\text{mL Titrant} \times \text{N Titrant}) \times 16000}{\text{g of Sample}}$$

$$\text{mg/Kg } S^{2-} \text{ (Back Impinger)} = \frac{(\text{mL } I_2 \times \text{N } I_2) - (\text{mL Titrant} \times \text{N Titrant}) \times 16000}{\text{g of Sample}}$$

$$\text{mg/Kg } S^{2-} \text{ (Sample)} = \text{mg/Kg } S^{2-} \text{ (Front Impinger)} + \text{mg/Kg } S^{2-} \text{ (Back Impinger)}$$

Note: The derived quantification in mg/Kg units does not take into account the moisture content of the sample. To report in mg/Kg (dry weight) units, use the following equation:

$$\text{mg/Kg } S^{2-} \text{ (Dry Weight)} = \frac{\text{mg/Kg } S^{2-} \text{ (Wet Weight)} \times 100}{\text{\% Solids in Sample}}$$

AVS is additionally reported in terms of umoles per gram. To report umole/gram units, use the following equation:

umoles/g S^{2-} = mg/Kg S^{2-} x 1Kg/1000g x 1umole/0.032mg = mg/Kg S^{2-} x 0.03125

12.3 SEM Calculation

For each trace metal:

$$\text{mg/Kg (Sample)} = \frac{\text{ug/L in Filtrate x 0.250L Filtrate x 1mg/1000ug x1000g/Kg}}{\text{g of sample}}$$

Note: The derived quantification in mg/Kg units does not take into account the moisture content of the sample. To report in mg/Kg (dry weight) units, use the following equation:

$$\text{mg/Kg (Dry Weight)} = \frac{\text{mg/Kg (Wet Weight)} \times 100}{\text{\% solids in sample}}$$

SEM is the additive total of all trace metals concentrations included in the evaluation. SEM is additionally reported in terms of umoles per gram. To report umoles/gram units for those metals typically included in the evaluation, use the following equations:

$$\text{umoles/g Cd} = \text{mg/Kg Cd} \times 1\text{Kg}/1000\text{gm} \times 1\text{umole}/0.112411\text{mg} = \text{mg/Kg Cd} \times 0.008896$$

$$\text{umoles/g Cu} = \text{mg/Kg Cu} \times 1\text{Kg}/1000\text{gm} \times 1\text{umole}/0.063546\text{mg} = \text{mg/Kg Cu} \times 0.01574$$

$$\text{umoles/g Hg} = \text{mg/Kg Hg} \times 1\text{Kg}/1000\text{gm} \times 1\text{umole}/0.20059\text{mg} = \text{mg/Kg Hg} \times 0.004985$$

$$\text{umoles/g Pb} = \text{mg/Kg Pb} \times 1\text{Kg}/1000\text{gm} \times 1\text{umole}/0.2072\text{mg} = \text{mg/Kg Pb} \times 0.004826$$

$$\text{umoles/g Ni} = \text{mg/Kg Ni} \times 1\text{Kg}/1000\text{gm} \times 1\text{umole}/0.0586934\text{mg} = \text{mg/Kg Ni} \times 0.01705$$

$$\text{umoles/g Zn} = \text{mg/Kg Zn} \times 1\text{Kg}/1000\text{gm} \times 1\text{umole}/0.06539\text{mg} = \text{mg/Kg Zn} \times 0.01529$$

12.4 SEM:AVS Ratio

$$\frac{\text{SEM}}{\text{AVS}} = \frac{\text{SUM of Metals (umoles/g)}}{\text{AVS (umoles/g)}}$$

For those metals that are not in a divalent state as the sulfide salt, then the calculation needs to be modified to account for the stoichiometry. If silver were to be included in the evaluation, the umoles/gram result for silver would need to be multiplied by a factor of 2 prior to the summation of the results of the trace metals. If antimony were to be included, a factor of 2/3 would be applied to the umoles/gram result for antimony prior to the summation.

12.5 LCS Percent Recovery (%R):

$$\%R = \frac{SR}{SA} \times 100\%$$

Where:
SR=Sample Result
SA=Concentration of Spike Added

12.6 MS Percent Recovery:

$$\text{MS Recovery}(\%) = \frac{\text{SSR - SR}}{\text{SA}} \times 100\%$$

Where:
SSR=Matrix Spike Result
SR=Sample Result
SA=Concentration of Spike Added

12.7 Relative Percent Difference (RPD):

$$\%\text{RPD} = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} \times 100$$

Where:
D1 = Sample result
D2 = Duplicate result

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Review the samples, standards and QC samples against the performance criteria given in section 9.0 for Quality Control. If the results do not fall within the established limits or criteria, corrective action is required as contained in Table 1. If corrective action is not taken or unsuccessful, the situation should be documented and reported in the project narrative. All data that does not meet established criteria must be flagged with the appropriate data qualifier and noted in the project narrative.

14.0 METHOD PERFORMANCE

14.1 A demonstration of analyst capability (IDOC) is required prior to use of this SOP and any time there is a significant change in instrument type, personnel or test method. IDOC procedures are further described in laboratory SOP LP-QA-011, *Employee Training*.

14.2 A Method Detection Limit (MDL) Study is performed at initial method set-up and subsequently once per 12 month period. The procedure and acceptance criteria for MDL studies are given in laboratory SOP LP-QA-005, *Method Detection Limits & Instrument Detection Limits.* MDL studies are kept on file by the QA Department.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Waste is disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.” The laboratory procedures for waste management are described in SOP LP-LB-001HAZWD.

15.2 Hazardous waste is accumulated in satellite containers located in the work area. Each

satellite container is labeled “Hazardous Waste” along with the name of the waste category. The Hazardous Waste Coordinator or designee routinely empties the satellite containers and transfers the waste to the hazardous waste storage room.

15.3 Waste Streams generated by this method

- Acidic waste – 55 gallon acid waste drum

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REVISION HISTORY

16.1 Title page: Updated to reflect current management team.

16.2 Section 1.2: Lowered mercury RL from 0.125 mg/kg to 0.025 mg/kg

16.3 Section 7.1: Added deaeration step for reagent water, hydrochloric acid, and impinger solution.

16.4 Section 7.1: Removed zinc acetate solutions and added 0.5N sodium hydroxide as the impinger solution.

16.5 Section 11.1: Changed zinc acetate solution for impingers to sodium hydroxide.

16.6 Section 16.0: Renamed Revision History

16.7 Section 17.0: Renamed References

16.8 Section 18.0: This section was added.

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994, Final Update IIB, January 1995, Final Update III, December 1996.

17.2 Standard Method for the Examination of Water and Wastewater, 19th edition, Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds, American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C., 1995.

17.3 USEPA Office of Water, Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment, December 1991.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Primary Materials Used, Exposure Limits and Hazards
18.2 Table 2: QC Summary and Recommended Corrective Action

Table 1: Primary Materials Used, Hazards & Exposure Limits

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Iodine	Corrosive Oxidizer Poison	0.1 ppm-Ceiling	Vapors severely irritate and can burn the mucous membranes and respiratory tract. Liquid contact may cause blistering burns, irritation, and pain. Vapors may be severely irritating to the skin. Vapors are severely irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous, it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Sulfide	Corrosive	10ppm - TWA 15 ppm-STEL	Will form Hydrogen Sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing, and, in extreme cases, pulmonary edema and possible death. Contact with the skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Contact will cause inflammation and tearing, and severe contact can cause permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 2 Quality Control Criteria and Corrective Action

QC Sample	Frequency	Acceptance Limits	Corrective Action
Method Blank	Each batch of 20 samples or less.	< Reporting Limit < ½ RL (DoD only)	Re-analyze, if still above re-prepare and re-analyze batch.
LCS	Each batch of 20 samples or less.	85-115%	The LCS is used to assess the proficiency in performing the method. Evaluate any LCS outside limits. Re-evaluate the calibration curve, and, if warranted, re-calibrate and re-analyze. Otherwise,
Matrix Spike (MS)	Every 20 samples	85-115%	The MS is used to assess the effect of the sample matrix on the accuracy of the method. Evaluate any MS outside limits and note as a non-conformance if a matrix effect is indicated.
Matrix Duplicate (MD)	Every 20 samples	RPD $\leq$ 20	The MD is used to assess the effect of the sample matrix on the precision of the method. Evaluate any MD outside limits and note as a non-conformance if a matrix effect is indicated.

Appendix A: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently, under the same laboratory conditions, also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL must be minimally at or above the MDL.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Acid Volatile Sulfides (S^{2-}): the sulfide present in the sample that is released under the test conditions of the method.

STANDARD OPERATING PROCEDURE
PARTICLE-SIZE ANALYSIS OF SOILS
ASTM D422-63
Applicable Matrix: Soil

APPROVAL SIGNATURES

Laboratory Director:	Christopher A. Ouellette	Date: August 5, 2005
QA Manager:	Kirstin L. McCracken	Date: August 5, 2005
Department Manager:	William S. Cicero	Date: August 5, 2005
Techncial Director: Geotechnical Laboratoy	Matthew R. Duquette	Date: August 5, 2005

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1.0 SCOPE AND APPLICATION

1.1. This SOP describes the laboratory procedure for the determination of particle size distribution in soil samples that contain sand, silt, clay and gravel.

2.0 SUMMARY OF METHOD

2.1. A soil sample submitted for particle size analysis is prepared according to laboratory SOP LM-SL-D421 *Dry Preparation of Soil for Particle Size Analysis* or LM-SL-D2217 *Wet Preparation of Soil Samples for Particle Size Analysis*. Particles greater than 75um (gravels to fine sands) are determined by sieve analysis while particles less than 75um (silts and clays) are determined by sedimentation using a hydrometer followed by sieve analysis.

After wet or dry sample preparation, the sample is passed through No.10 sieve. The particles retained on the No.10 sieve (greater than 2.00mm) are further separated by sieve analysis. A portion of the sample that passed through the No.10 sieve is transferred to a glass sedimentation cylinder to which distilled water has been added. Seven hydrometer readings are taken over 24 hours. After the final hydrometer reading, the suspension is rinsed over a No. 200 (75 um) sieve, dried, and further separated by sieve analysis.

Particle size determinations for each sieve measurement and hydrometer reading are calculated and corrected for hygroscopic moisture and specific gravity. Unless a separate analysis for specific gravity is requested, the specific gravity is s assumed to be 2.65.

2.2. This procedure is based on ASTM D422-63.

3.0 DEFINITIONS

Not Applicable

4.0 INTERFERENCES

Not Applicable

5.0 SAFETY

5.1. Care should be taken to avoid exposure to the sample matrix since all environmental samples are potentially hazardous. Protective clothing, eye protection and disposable gloves should be worn when handling samples. All laboratory personnel must be familiar with the environmental health and safety plan described in the STL Chemical Safety Manual.

6.0 EQUIPMENT AND SUPPLIES

6.1. Top-Loading Balance sensitive to 0.01 g

6.2. Mechanical Stirring Apparatus and Dispersion Cup

6.3. Sedimentation Cylinder(s) 1000 mL

6.4. Hydrometer: ASTM 151H in specification E 100.

6.5. Thermometer: Accurate to 0.5°C

6.6. Mortar and Rubber Tipped Pestle

6.7. Sieves of the following size(s):

3.0 in (75.00mm)	No. 20 (850.0um)
2.0 in (50.00mm)	No. 40 (425um)
1.5 in (37.50mm)	No. 60 (250.0um)
1.0 in (25.00mm)	No. 80 (180.0um)
3/4 in (19.00mm)	No. 100 (150.0um)
3/8 in (9.50mm)	No. 200 (75.0um)
No. 4 (4.75mm)	
No. 10 (2.00mm)	

6.8. Oven with temperature range of 60° C to 110° C

6.9. Timing Device with second hand and capable of counting up to 25 hours

6.10. Stainless steel spatulas, spoons, metal and bristle brushes

6.11. Ro-tap machine

7.0 REAGENTS AND STANDARDS

7.1. Reagents

Deionized (DI) Water: Milli-Q System

Sodium Hexametaphosphate Solution: Combine 2940 g of DI water with 120 g of sodium hexametaphosphate in an appropriate container. Mix until the solution is homogeneous. Assign an expiration date of 30 days from date of preparation.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1. At least 500 grams of soil sample should be collected in glass or polyethylene jars. Immediately following collection the sample should be sealed and cooled to 4°C in order to preserve the moisture content of the sample.

8.2. Samples are stored from the time of receipt in the laboratory until 30 days after delivery of the reconciled data package report. Unless otherwise specified by a federal, state or

client-specific protocol, samples are disposed of after 30 days in a manner that complies with all applicable regulations.

9.0 QUALITY CONTROL

Not Applicable

10.0 CALIBRATION AND STANDARDIZATION

10.1. Calibrate the balance on each day of use, prior to use.

10.2 Calibrate the hydrometers every two years following the procedure given in LM-SL-001.

11.0 PROCEDURE

11.1 Sample Preparation

Prepare the sample following either laboratory SOP LM-SL-D421 (Dry Preparation) or LM-SL-D2217 (Wet Preparation).

11.2 Sample Analysis

11.2.1 Hydrometer Test

Transfer the sample/sodium hexametaphosphate mixture into a dispersion cup ensuring a quantitative transfer using DI water. Fill the dispersion cup ~half full with DI water. Mix the sample for one minute using the immersion blender.

Pour the contents of the dispersion cup through a #10 sieve into a 1000 mL sedimentation cylinder (1000 mL graduated cylinder). Rinse the cup with DI water, to ensure that the entire sample is transferred to the sedimentation cylinder.

Add DI water to the sedimentation cylinder until the volume is 1000 mL then cover the cylinder with a sheet of parafilm. Allow the sample to stabilize to ambient temperature.

Transfer the material retained on the No. 10 sieve to a labeled medium-size aluminum dish, and place the aluminum dish into an oven maintained at a temperature of 105ºC for a minimum of 16 hours.

After up to 12 sedimentation cylinders have been prepared, ensure that each cylinder is filled to the reference line with DI water, covered with parafilm, and that there is sufficient clean DI water available to rinse the hydrometer.

Record the ID of the hydrometer that you intend to use. Record the start time and set the timer for elapsed time.

Use the hydrometer reading table used to perform the activities as indicated (shake, place or read) for each 1000 mL cylinder.

To shake, rotate the flask up and down for one minute approximating at least 60 turns (one turn upside down and then right side up constitutes two turns).

To take a reading, gently insert the hydrometer into the cylinder then wait ~ 20 seconds. Read the hydrometer at the top of the meniscus to the nearest 0.0005. Enter the hydrometer reading into the appropriate cell on the benchsheet. Clean the hydrometer by twisting and dropping into a clean DI water bath.

Insert a temperature sensor into the cylinder to the depth the hydrometer reached. Read the temperature to the nearest 0.5°C. Enter the temperature reading into the appropriate cell on the benchsheet. After reading, rinse the sensor in a DI water bath.

Take readings every 2, 5, 15, 30, 60, 240 and 1440 minutes. Record each reading on the benchsheet, then transfer this information into the appropriate cell of the EXCEL worksheet.

11.2.2 Large Sieves

Enter a brief description of the type of non-soil material (e.g.- sticks, grass, wood, plastic) in the “Description of >#10 particles” of the EXCEL worksheet in the cell labeled “Non-soil Material”.

Tare the balance and weigh each of the 3/4”, 3/8”, #4 and #10 sieves. Record the weight measurements in the EXCEL worksheet in the cells labeled “Sieves (Tares)”. Also weigh any larger sieves if necessary.

Stack the sieves then transfer the soil material retained on the #10 sieve into the sieve stack. Shake for 2 minutes. If there is greater than ~30 g of material, place the sieve stack into the Ro-tap machine and shake for 10 minutes.

Weigh each sieve along with the material retained on it. Enter these weight measurements in the “Sieve + Sample Weights” section of the Excel worksheet.

Determine the average hardness of the particles retained on the #10 sieve by dropping a hammer on the particle from a height of approximately one foot. Hardness qualifiers are hard, soft or brittle. Record the hardness qualifier in the “Description of >#10 Particles” section of the Excel worksheet.

Observe and record the shape of the particles in the “Description of >#10 Particles” section of the Excel worksheet. Shape qualifiers are well rounded, rounded, subrounded, subangular, and angular.

11.2.3 Small Sieves

When the hydrometer test is complete, transfer the soil from the sedimentation cylinder to a #200 wet wash sieve.

Wash the soil through the #200 sieve until the water from the bottom of the sieve runs clear. Carefully transfer the material retained on the sieve to a labeled 250 mL glass beaker.

Place the beaker into the oven. Dry at a temperature of 105ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

Gently mix the dried contents of the beaker with a rubber-tipped pestle to break any soil aggregates that may have formed during the drying stage.

Tare the balance and weigh each of the sieves between #20 and #200. Record the weight measurements in the EXCEL worksheet in the cells labeled “Sieves (Tares)”.

Transfer the dry sample into the sieve stack, ensuring that all material is transferred. Use hair or wire brushes to clean the beaker.

Place the sieve stack on the Rotap machine and shake for ten minutes.

Weigh each sieve along with the material retained on it. Enter these weight measurements in the “Sieve + Sample Weights” section of the Excel worksheet.

Determine particle size using the following formula.

12.0 CALCULATIONS

12.1. Sample Used (SU)

Wet Method

$$SU = (pan + wet\ sample - pan) \otimes PS$$

Where:

PS = Percent solids

Note: for hydrometer SU, subtract the dry weight of any material retained on the No. 10 sieve.

Dry Method

$$SU = (pan + dry\ sample - pan) - (pan + non - soil\ material - pan) \otimes HMCF$$

Where:

HMCF = Hygroscopic moisture correction factor

12.2 Sieve Analysis (Percent Finer = PF)

Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

12.5 Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Viscosity at sample temperature, poises
Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8)+10.5 and 264.5 = (10.5-2.3)/0.031
Time, minutes = Time of hydrometer reading from beginning of sedimentation
Sqrt - square root
SG - Specific Gravity of soil
Viscosity - is the resistance of a liquid to flow

12.6 Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

Where:
Constant = (100,00/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))
Intercept = (low temp. reading -1) - (low temp. * slope)

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.0. Complete the sample preparation benchsheet and EXCEL spreadsheet. Document any problems encountered during sample analysis so they may be properly addressed in the project narrative. Perform primary and secondary data review following the guidance given in laboratory SOP for Data Review.

14.0 METHOD PERFORMANCE

Not Applicable

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1. The laboratory optimizes technology to minimize pollution and reduce the production of hazardous waste whenever possible.

15.2. The laboratory procedures for waste management comply with applicable federal, state and local regulations and are described in SOP LP-LB-001HAZWD.

16.0 REFERENCES

16.1. Standard Test Method for Particle-Size Analysis of Soils, ASTM D422-63, Volume 04.08 Soil and Rock, American Society for Testing and Materials, Philadelphia, Pa., 1998.

17.0 REVISION HISTORY

17.1 Procedures to separate the non-soil particles from the sample were removed.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Hydrometer Reading Table

Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)

Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)	Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)
0:00	Shake	1		1:01	Read	10	5
0:01	**Place**	**1**		**1:02**	Shake	11	
0:01	**Shake**	**2**		**1:03**	Place	11	
0:02	Place	2		1:04	Read	9	15
0:03	Read	1	2	1:05	Read	11	2
0:04	Read	2	2	1:06	Read	7	31
0:06	Read	1	5	1:07	Read	3	58
0:07	Read	2	5	1:08	Read	11	5
0:08	Shake	3		**1:09**	Shake	12	
0:09	**Place**	**3**		**1:10**	Place	12	
0:09	**Shake**	**4**		1:11	Read	10	15
0:10	Place	4		1:12	Read	12	2
0:11	Read	3	2	1:13	Read	4	63
0:12	Read	4	2	1:14	Read	8	32
0:14	Read	3	5	1:15	Read	12	5
0:15	Read	4	5	1:18	Read	11	15
0:16	Read	1	15	1:19	Read	9	30
0:17	Read	2	15	1:21	Read	5	60
0:20	Shake	5		1:25	Read	12	15
0:21	Place	5		1:26	Read	10	30
0:23	Read	5	2	1:27	Read	6	59
0:24	Read	3	15	1:33	Read	11	30
0:25	Read	4	15	1:34	Read	7	59
0:26	Read	5	5	1:41	Read	12	31
0:27	Shake	6		1:42	Read	8	60
0:28	Place	6		1:52	Read	9	63
0:30	Read	6	2	1:53	Read	10	57
0:31	Read	1	30	2:06	Read	11	63
0:32	Read	2	30	2:07	Read	12	57
0:33	Read	6	5	4:17	Read	1	256
0:34	Shake	7		4:18	Read	2	256
0:35	Place	7		4:19	Read	3	250
0:36	Read	5	15	4:20	Read	4	250
0:37	Read	7	2	4:21	Read	5	240
0:38	Read	3	29	4:22	Read	6	234
0:39	Read	4	29	5:00	Read	7	265
0:40	Read	7	5	5:01	Read	8	259
0:41	Shake	8		5:02	Read	9	253
0:42	Place	8		5:03	Read	10	247
0:43	Read	6	15	5:04	Read	11	241
0:44	Read	8	2	5:05	Read	12	235
0:47	Read	8	5	24:01	Read	1	1440
0:48	Shake	9		24:02	Read	2	1440
0:49	Place	9		24:03	Read	3	1434
0:50	Read	7	15	24:04	Read	4	1434
0:51	Read	9	2	24:05	Read	5	1424
0:52	Read	5	31	24:06	Read	6	1418
0:54	Read	9	5	24:07	Read	7	1412
0:55	Shake	10		24:08	Read	8	1406
0:56	Place	10		24:09	Read	9	1400
0:57	Read	8	15	24:10	Read	10	1394
0:58	Read	10	2	24:11	Read	11	1388
0:59	Read	6	31	24:12	Read	12	1382
1:00	**Read**	**1**	**59**				
1:00	**Read**	**2**	**58**				

Source: Laboratory Prepared Reference Document

STANDARD OPERATING PROCEDURE
HOMOGENIZATION AND EXTRACTION OF BIOTA & TISSUE BY TISSUMIZER

Applicable Matrices: Tissue and Biota

APPROVAL SIGNATURES

Laboratory Director: Michael F. Wheeler, Ph.D. Date: 5/20/05

QA Manager: Kirstin L. McCracken Date: 5/20/05

Department Manager: Jon K. Zygmuntowicz Date: 5/20/05

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1.0 SCOPE AND APPLICATION

1.1. This SOP describes the laboratory procedure for the extraction of biota and tissue samples by tissumizer in preparation for analysis by a variety of chromatographic procedures.

2.0 SUMMARY OF METHOD

2.1 Homogenization

Tissue samples are homogenized using a titanium blade homogenizer. Biota samples are homogenized using stainless steel or ceramic knives. The homogenized sample(s) are transferred to labeled glass jars and stored in a freezer maintained at a temperature of -15ºC (±5ºC) in preparation for extraction.

This procedure was derived from the Comprehensive Descriptions of Trace Organic Analytical Methods given in the Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, National Status and Trends Program for Marine Environmental Quality.

2.2 Extraction

A portion of homogenized sample is mixed with anhydrous sodium sulfate then macerated for 3 minutes in an appropriate extraction solvent using the Tissumizer. The extraction is repeated two more times with fresh portions of extraction solvent. After extraction, the extracts combined in a KD flask in preparation for concentration to an appropriate final volume. Percent lipids are determined following procedures given in *l*aboratory *SOP LM-OP-LIPIDS and e*xtract cleanup is performed when appropriate.

This procedure is based on method GERG Trace Organic Contaminant Analytical Techniques published in NOAA Technical Memorandum NOS Orca 71, Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, Comprehensive Descriptions of Trace Organic Analytical Methods, July 1993.

3.0 DEFINITIONS

3.1. Biota: flora and fauna. For this SOP, all reference to "biota" refers to plant material.

3.2. Tissue: an aggregate of cells usually of a particular kind together with their intercellular substance that form one of the structural materials of a plant or animal. For this SOP, all reference to "tissue" refers to structural materials from an animal.

3.3. A list of general terms and definitions is provided in Appendix A.

4.0 INTERFERENCES

4.1. Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference. All glassware must be cleaned in accordance with laboratory SOP *LP-OP-Glass* and rinsed with acetone prior to use.

5.0 SAFETY

5.1. Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2. Safety Concerns or Requirements

Protective clothing such as a lab coat, safety glasses and latex gloves must be worn while performing this procedure.

5.3. Primary Materials Used

Table 1, Section 17.0 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1. Tissuemizer:

- Kuderna-Danish Apparatus:

  Concentrator tube - 10mL graduated (AMK KD-0018 or equivalent)
  Evaporation flask - 500mL (AMK KD-0015 or equivalent)

- Snyder column- Three Ball Macro (AMK KD-0011); Two Ball Micro (AMK KD-0013)

- Heating Mantle, rheostat controlled for water bath capable of temperature control (± 5°C).

- Boiling chips - silicon carbide, approximately 10/40 mesh, solvent extracted with methylene chloride.

- Disposable glass Pasteur pipettes and bulb.

- Fisher Scientific PowerGen 700 Tissumizer equipped with a 20 mm x 195 Generator probe.
- Balance - capable of measuring 100 g to ± 0.01 g
- Filter Funnels – 100 mm diameter for filtration/drying.
- Filter paper - No. 541 Whatman 18.5 cm, or equivalent.
- Beakers – 400 mL.
- Stainless steel or Teflon spatulas.
- Adjustable pipette
- 8 and 16 mL comparison vials.
- Vials and caps: 1.8, 4, 8, and 16 mL with Teflon lined septa and screw caps.

6.2 Homogenizer

- Cutting Board- High density polyethylene 16X23"
- Analytical Balance
- Omni- Mixer/Homogenizer equipped with 55 mm Titanium Blade or equivalent.
- Waring-Commercial stainless steel blender or equivalent.
- Cuisinart Food Processor
- Stainless steel/Ceramic knives
- Glass Jars, wide mouth; 125 mL-1000 mL
- Teflon & Stainless Steel Spatulas
- Paper towels
- Nitrile gloves

7.0 REAGENTS AND STANDARDS

7.1. Reagents

Organic-free reagent water

Liqui-Nox Detergent

Sodium Sulfate (granular, anhydrous), Na2SO4. J.T. Baker. Bake in an oven maintained at a temperature of 400°C for 4 hours prior to use.

Methylene Chloride, CH2C12- J.T. Baker, Pesticide quality or equivalent

Hexane, C6H14- J.T. Baker, Pesticide quality or equivalent

Acetone, (CH3)2CO- J.T. Baker, Pesticide quality or equivalent

7.2. Stock Standard Solutions

Stock standard solutions are purchased pre-certified from commercial vendors. Working standard solutions are prepared in the laboratory by diluting a volume of standard or reagent solution into an appropriate medium. The preparation of standard solutions is documented in a logbook designated for this purpose.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT & STORAGE

8.1. Tissue and biota samples should be collected in glass jars. Immediately following collection, biota samples should be iced to a temperature of 4°C (±2°C) and tissue samples should be frozen and maintained at a temperature of -15°C (±5°C) until the time of homogenization. After homogenization is complete, all samples must be stored in a freezer maintained at a temperature of -15°C (±5°C).

8.2. Tissue and biota samples should be extracted within 7 days after the sample was thawed or removed from refrigerated storage. The sample should be returned immediately to the freezer after extraction. As long as the samples are frozen, a six-month holding time will apply.

8.3. Unless otherwise specified by a federal, state or client-specific protocol, samples are stored from the time of receipt in the laboratory until 30 days after delivery of the reconciled data package report, at which time the samples are disposed of in a manner that complies with the applicable regulations.

9.0 QUALITY CONTROL

9.1. A method blank (MB) and lab control sample (LCS) is performed with each preparation batch of 20 field samples or less. Matrix spikes and sample duplicates are performed when requested by the client.

9.2. All field samples, QC items, and matrix spikes are fortified with surrogate spike prior to extraction.

9.3. The LCS and the matrix spikes are fortified with spike solution, which includes all analytes of interest prior to extraction.

9.4. The criteria used to assess quality control samples are described in the laboratory SOP for the determinative analysis test method.

10.0 CALIBRATION AND STANDARDIZATION

10.1. Calibrate the analytical balance on each day of use, prior to use with at least 3 Class S weights that braket the range of use.

10.2. Calibrate the mechanical volumetric pipettes each day of use, prior to use, following the approved laboratory standard operating procedure.

10.3. Perform periodic maintenance on the Tissumizer's Generator Probe as necessary. Maintenance may include but is not limited to the replacement of Teflon bearings and rotor shafts when a loud squealing noise is heard. Refer to the PowerGen 700 Homogenizer Instruction Manual for further guidance and for the manufacturer's recommended maintenance program.

11.0 PROCEDURE

11.1 Homogenization of Tissue Samples

Prior to use and between each sample, wash all equipment with Liqui-Nox detergent and hot water. Rinse the equipment with DI water and allow to air dry prior to use. After each sample, disassemble the blender, wash thoroughly, and rinse the homogenizer blade with reagent water, acetone and methylene chloride prior to use.

To ensure the equipment is clean, prepare equipment blank by transferring 50 mL of reagent water to the piece of equipment that will be used for homogenization. Blend the reagent water for two minutes and transfer to a labeled glass jar.

Remove the samples from storage and allow sufficient time for them to completely thaw.

Select a glass jar large enough to accommodate the entire sample. Label the jar with the sample's lab ID and place the jar on the analytical balance. Tare the balance. Put on a pair of nitrile gloves, and using your hands, remove the tissue sample from the storage container and place in the labeled jar. Record the initial weight of the sample on the bench sheet.

Remove the sample and place on a pre-cleaned cutting board. Cut the sample into 1-3" sections using a stainless steel knife (for samples designated for organic analyses) or a ceramic knife (for samples designated for metals analyses). Retare the balance and place the sectioned sample in the labeled jar. Insert the titanium blade into the jar and homogenize the sample at 2000-4000 RPM for 2 minutes or until the sample becomes a slurry. Manual mixing with a stainless steel or Teflon spatula (stainless steel for organic analyses and Teflon for metals) may be required to insure complete homogenization. Remove the blade from the sample jar and scrape any remaining sample from the blade into the labeled jar. Record the final weight measurement on the bench sheet.

Note: Samples that are greater than 12 inches or samples with weight measurements that exceed 1-2 pounds should be homogenized in a stainless steel blender or food processor prior to blade homogenization.

11.2 Homogenization of Biota Samples

Prior to use and between each sample, wash all equipment with Liqui-Nox detergent and hot water. Rinse the equipment with DI water and allow to air dry prior to use.

To ensure the equipment is clean, prepare equipment blank by transferring 50mL of reagent water to the piece of equipment that will be used for homogenization. Blend the reagent water for two minutes and transfer to a labeled glass jar.

Remove the samples from storage. Select a glass jar large enough to accommodate the entire sample. Label the jar with the sample's lab ID and place the jar on the analytical balance. Tare the balance. Remove the biota sample from the storage container and place in the labeled jar. Record the initial weight of the sample on the bench sheet.

Remove the sample and place on a pre-cleaned cutting board. Slice the material into very fine sections using a stainless steel (organics) or ceramic (metals) knife. Re-tare the balance and transfer the sample to the labeled jar. Record the final weight measurement on the bench sheet.

11.3 Extraction Procedure

Clean glassware prior to use following the procedure given in laboratory SOP *LP-OP-Glass.* Label samples and QC items clearly and unambiguously during each step of the extraction procedure. Solvents will erase grease pens and "sharpie ink" caution must be taken to ensure that the labels are not obliterated during the procedure.

Assemble a KD apparatus set-up and prepare a glass funnel for each sample to be extracted. Fold a 185 mm Whatman® 54 filter into quarters and place a filter in each funnel. Fill each funnel ~3/4 full with purified granular anhydrous sodium sulfate. Rinse the funnel with ~30 mL acetone and discard the solvent rinse. Place a prepared funnel onto each K-D setup.

Assemble the Tissumizer by attaching the 20 mm x 195 mm-generator probe to the Tissumizer motor. Place the Tissumizer in the fume hood and attach to the aluminum staging using clamps. Clean the Tissumizer prior to use by running the generator probe for 10 seconds in a 400 mL beaker filled with ~ 100 mL of reagent water. Discard the reagent water and repeat with another aliquot of reagent water. Repeat the cleaning step two more times each with ~100 mL of acetone and then ~100 mL of methylene chloride.

Open the sample container and inspect the sample. Mix the sample using a stainless steel or Teflon spatula. Place a labeled 400 mL beaker onto the top-loading balance and depress the "tare" button. Weigh the appropriate amount of sample within +/- 0.05 g into a labeled 400 mL beaker and repeat for all samples. Transfer two additional

aliquots of the sample selected for the MS and MSD into labeled 400 mL beakers.Transfer the same weight of sodium sulfate each into labeled 400 mL beakers to serve as the method blank (MB) and laboratory control sample (LCS).

Add sufficient volume of granular sodium sulfate to each sample and mix thoroughly with a stainless steel spatula until a free-flowing mixture is formed.

Add the appropriate volume of surrogate spike to each sample and QC item.

Add the appropriate volume of spike solution to the laboratory control samples and the MS/MSD.

Add 100 mL of the appropriate extraction solvent to each beaker. Use 1:1 $MeCl_2$/Acetone for samples to be analyzed by GC/MS and 1:1 Hexane/Acetone for GC/ECD.

Immerse the generator probe in the first sample beaker so that it is approximately ½" into the extraction solvent. Turn on the Tissumizer. Adjust the speed on the motor until the solvent begins to vortex in the beaker, but does not splash out of the beaker. Extract the sample for 3 minutes. During extraction move the beaker in a circular motion to ensure that the entire sample is subject to extraction. Remove the beaker and decant the extraction solvent into the sample's corresponding funnel and K-D apparatus. Repeat the extraction 2 more times with ~100 mL of extraction solvent.

Rinse the funnel with ~30 mL of extraction solvent and allow the solvent to completely drain into the K-D apparatus. Remove the funnel from the K-D apparatus and discard the contents of the funnel. Clean the generator probe and repeat the extraction for each sample.

Concentrate the extracts following the procedure given in section 11.4 in preparation for percent lipids determination and extract cleanup. After concentration and prior to extract cleanup, set aside a 1 mL aliquot of the concentrated extract and determine the percent lipids following procedures given in laboratory SOP *LM-OP-LIPIDS*.

Perform extract cleanup as appropriate following procedures given in laboratory SOPs *LM-OP-Cleanup* and *LM-OP-GPC.* After cleanup concentrate the extracts following the procedure given in section 11.5. Enter the extraction data into the Organic Extraction Module in LIMS. Assemble the bench sheet and any associated paperwork and submit the extracts to the supervisor for a final project check. After review is complete, relinquish the extracts to the appropriate analytical department and place in the refrigerated storage area.

Note: Immediately following concentration, all sample extracts must be stored in a refrigerator maintained at a temperature of 4ºC (±2°C) in order to maintain thermal preservation.

11.4 Extract Concentration

Concentrate PCB extracts to 3~4 mL in hexane in preparation for acid cleanup. After acid cleanup, concentrate the extracts to 10.0 mL in hexane.

Concentrate GC/MS and pesticide extracts to 10 mL in methylene chloride in preparation for GPC cleanup. After GPC, concentrate the SVOA extracts to 0.5 mL in methylene chloride in preparation for GC/MS analysis. Concentrate the pesticide extracts to 5.0 mL in hexane and remove a 1 mL aliquot and perform Florisil cleanup. After Florisil cleanup, concentrate the extracts 1 mL in hexane.

11.4.1 Macro Snyder Column K-D

Add one or two clean boiling chips to the KD flask and attach a three-ball Snyder column.

Pre-rinse the Snyder column by adding ~10 mL of acetone to the top of the column.

Place the K-D apparatus on a hot water bath (15-20° C above the boiling point of the solvent) 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.

Attach the solvent vapor recovery glassware to the Snyder column of the K-D apparatus following manufacturer's instructions.

Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood.

When the volume of extract is approximately <10 ml, perform the solvent exchange as necessary and continue to concentrate the extract to less than <10 ml. If solvent exchange is not required, proceed to the next step.

Remove the K-D apparatus from the water bath and allow it to cool and drain for at least 10 minutes.

Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of the appropriate solvent.

Concentrate the sample to the desired final volume using the micro synder K-D technique.

11.4.2 Micro-Snyder Column K-D

Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column.

Place the concentrator tube into the water batch so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the concentrator tube and/or the temperature of the water bath to ensure that concentration of the extract is complete within 5-10 minutes.

Continuously monitor the distillation process to ensure sample extracts do not evaporate too quickly. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with solvent.

When the volume of liquid in the concentrator tube reaches 0.5-1.0 mL, remove from the hot water bath.

***Caution**: Extracts designated for 8270 PAH should not be allowed to concentrate to less than 1mL or volatilization of target compounds will occur.*

Adjust the extract to the appropriate volume in preparation for cleanup or analysis. For final extract volumes ≤ 1.0 mL, the extract volume is adjusted using a calibrated, graduated concentrator tube. For final extract volumes ≥1.0 mL the extract volume is adjusted using a comparison vial.

Transfer the extract into a labeled Teflon lined screw or crimp-top vial.

If extract cleanup is not required, complete the extraction bench sheet. Enter the extraction data into the Organic Extraction Module in LIMS. Attach associated paperwork and submit extracts to the department supervisor for review. After review is complete, relinquish the extracts to the appropriate analytical department.

If extract cleanup is required, complete the extraction bench sheet and place the paperwork in the location designated for work scheduling of extract cleanup.

11.4.3 Nitrogen Blow Down

Nitrogen blow down should be used when concentration of the extract by the K-D technique is difficult due to high extract viscosity. Place the concentrator tube in a warm bath (65°C) and apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate dry.

12.0 CALCULATIONS

Not Applicable

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1. The extraction bench sheet is completed by the analyst(s) that performed the procedure and reviewed by the Department Supervisor. Problems encountered during the

extraction process are documented using a nonconformance report that records the root-cause, action taken, and the result of action taken. The QC acceptance criteria are given in the laboratory SOP for the analytical method. Results that do not meet the minimum acceptance criteria are flagged using data qualifiers and a description of the outage is written in the case narrative provided with the data package report.

14.0 METHOD PERFORMANCE

14.1. An Initial Demonstration of Capability is required for each analyst before unsupervised performance of this method.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1. Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is carried out.

- Acidic Waste
- Solvent Waste
- Expired primary and working PCB standards
- Vials containing sample extracts

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waster storage room for future disposal in accordance with Federal, State and Local regulations, The procedures for waste management are further given in the laboratory SOP LP-LB-001 *Hazardous Waste.*

16.0 REFERENCES

16.1. NOAA Technical Memorandum NOS Orca 71, Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, Comprehensive Descriptions of Trace Organic Analytical Methods, July 1993.

16.2. *Comprehensive Descriptions of Trace Organic Analytical Methods*, Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, National Status and Trends Program for Marine Environmental Quality. NOAA Technical Memorandum NOS ORCA 71.

17.0 TABLES, DIAGRAMS, FLOWCHARTS

17.1 Table 1: Primary Materials Used

REVISION HISTORY

Revision 3: The procedures for extraction previously described in laboratory SOP LM-OP-Tissumizer were incorporated into this SOP. LM-OP-Tissumizer was made obsolete.

Table 1: Primary Materials Used

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: General Laboratory Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

STANDARD OPERATING PROCEDURE
STL BURLINGTON

PERCENT LIPID DETERMINATION

Applicable Matrix: Biological Tissue

APPROVAL SIGNATURES

William S. Cicero
Laboratory Director
Date: December 20, 2006

Kirstin L. McCracken
Quality Assurance Manager
Date: December 20, 2006

Jacob L. Bailey
Department Manager
Date: December 20, 2006

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the laboratory procedure for the determination of percent lipids in biological tissue.

2.0 SUMMARY OF METHOD

2.1 A 1 mL aliquot of extract is evaporated to dryness and weighed to a constant weight. The residual weight of the dried aliquot is used to calculate percent lipids.

2.2 This procedure is based on EPA SOP ASB P100, ASB E100 for the Determination of Organics in Fish and NOAA Technical Memorandum NOS ORCA 130 Method for the Determination of Percent Lipid in Tissue, Sampling and Analytical Methods of the National Status and Trends Program Mussel Watch Project: 1993-1996 Update.

The following exceptions to the NOAA reference method are noted:

- The laboratory does not include the dry weight determination of the tissue sample in the percent lipids calculation.
- The tissue sample may be extracted with hexane extract instead of dichloromethane when hexane is an appropriate extraction solvent for the determinative method.
- The percent lipids are determined from the extract using a gravimetric procedure without the additional filtering or concentration steps.

3.0 DEFINITIONS

3.1 Definitions are included in Appendix A.

4.0 INTERFERENCES

4.1 Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference. All glassware must be cleaned in accordance with laboratory SOP LP-OP-Glass and rinsed with acetone prior to use.

5.0 SAFETY

5.1 Employees must be trained on and adhere to the policies and procedures for safety in the Corporate Safety Manual and this document.

5.2 Specific Safety Concerns or Requirements

None

5.3 Primary Materials Used

Table 1, Section 18.0 lists those materials that are part of this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 EQUIPMENT AND SUPPLIES

6.1 Aluminum Weigh Boat(s)

6.2 Analytical Balance

6.3 Adjustable Pipette(s)

6.4 Drying Oven

6.5 Stainless Steel Tongs

7.0 REAGENTS AND STANDARDS

7.1 Reagents

Anhydrous Sodium Sulfate- Purify for 4 hours at 400°C.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT & STORAGE

8.1 Sample extracts used for percent lipid determination must be protected from light and maintained at a temperature of 4(±2)°C.

8.2 Unless otherwise specified by client or regulatory program, after extraction samples and extracts are retained for 30 days then disposed of in accordance with applicable regulations.

9.0 QUALITY CONTROL

9.1 A method blank comprised of sodium sulfate is extracted with each batch of 20 or less samples and it is taken through the percent lipids determination in the same manner as sample extracts. The calculated percent lipids in the method blank should be <0.1%.

9.2 A duplicate sample should be performed with each extraction batch or at the frequency designated by the client. The relative percent difference (RPD) in the percent lipids determination between replicate samples should be less than or equal to 25%. If it is

not, the original and duplicate sample extracts should be re-weighed. If the RPD is still not within specification, the percent lipids procedure should be repeated using new extracts.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibrate the analytical balance and mechanical pipettes on each day of use, prior to use.

11.0 PROCEDURE

11.1 Percent Lipid Determination

Weigh an aluminum boat to the nearest 0.0001 g and record the weight measurement. Tare the balance and transfer 1 mL of the 10 mL extract (pre-cleanup) to the aluminum weigh boat. The extract should be filtered using a Whatman 0.45um Autovial Filter before concentration to 10.0 mL. Using tongs transfer the weigh boat to a drying oven maintained at a temperature of 104˚C (±2°C) and allow sufficient time for the solvent to evaporate to dryness. Allow the weigh boat/sample to cool to room temperature.

Re-weigh the weigh boat to the nearest 0.0001 g and record the weight measurement.

Calculate the percent lipids using the equation given in Section 12.0.

Evaluate the results of the method blank and sample duplicate against the criteria given in Section 9.0 Quality Control and perform corrective action as necessary.

12.0 CALCULATIONS

12.1 Percent Lipid Calculation

$$\%Lipids = \frac{RW}{AV} \otimes \frac{EV}{WS} \otimes 100$$

Where RW is the residue weight *(residue + weigh boat)* minus the weight of the weigh boat, AV is the volume of the aliquot used (mL), EV is the final extract volume (mL), and WS is the original weight of sample extracted (g).

12.2 Relative Percent Difference (RPD)

$$\frac{C_1 - C_2}{\left(\frac{C_1 + C_2}{2}\right)} \otimes 100$$

Where:
C_1 = Result of Parent Sample

C_2 = Result of Duplicate Sample

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.1 Primary Review: Review the bench sheet for correctness and completeness. Record any problems encountered during the process on the bench sheet or complete a nonconformance report, when necessary. Set aside the extracts and paperwork for secondary review.

13.2 Secondary Review: Review the bench sheet against the preparation worksheet to ensure the procedure performed is consistent with project specifications and this SOP.

13.3 For additional guidance regarding the laboratory's protocol and required elements for data review refer to laboratory SOP LP-QA -019 *Data Review*.

14.0 METHOD PERFORMANCE

14.1 This section is not applicable to this SOP.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Where reasonably possible technology changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this SOP and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this SOP is performed:

Organic Solvents - Satellite container: 55 gallon covered and vented drum.
Solid Waste - Satellite container: 5 gallon covered bucket in fume hood.

Transfer the waste stream(s) to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are further given in laboratory SOP LP-LB-001 *Hazardous Waste*

16.0 REVISION HISTORY REFERENCES

16.1 Changed Title page to reflect current management team.

16.2 Section 16: Added Revision History section.

17.0 REFERENCES

17.1 *SOP for the Determination of Organics in Fish.* SOP ID: ASB P100, Mod1.0 October 1990; ASB E100, Mod1.0, October 1990. USEPA Region 4 Science and Ecosystem Support Division, Analytical Support Branch.

17.2 *Determination of Percent Lipid in Tissue.* Geochemical and Environmental Research Group, Texas A&M University College Station, TX. NOAA Technical Memorandum NOS ORCA 130 National Status and Trends Program for Marine Environmental Quality Sampling and Analytical Methods of the National Status and Trends Program Mussel Watch Project: 1993-1996 Update

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Primary Materials Used
18.2 Appendix A: Terms and Definitions

Table 1: Primary Materials Used

Material	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[2] Exposure limit refers to the OSHA regulatory exposure limit.

Appendix A: Terms & Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

ARCADIS BBL

Attachment 1-3

KAR Quality Assurance Management Plan

KAR *Laboratories, Inc.*

ENVIRONMENTAL LABORATORY
QUALITY ASSURANCE MANUAL

November 27, 2006

KAR Laboratories, Inc.
4425 Manchester Road
Kalamazoo, MI 49001
269-381-9666
Fax: 269-381-9698
email: info@karlabs.com
Internet: http://www.karlabs.com

Environmental Laboratory
Quality Assurance Manual

Company Vision: Be a Top Performing Environmental Laboratory

Mission: Focus on the following key areas

Clients	*All resources are applied to satisfy or exceed clients' needs*
Employees	*The work environment provides a high degree of worker satisfaction*
Ethics	*All company and employee actions are legal and ethical*
Image	*KAR's image accurately reflects the company's commitment to excellence*
Profits	*KAR Laboratories is profitable for its shareholders and employees*
Quality	*Superior quality is provided in all aspects of performance*

Date of Issue:

Approval:

Vice President, Director of Laboratories: Michael J. Jaeger _____ Date: 11/27/06
Michael J. Jaeger

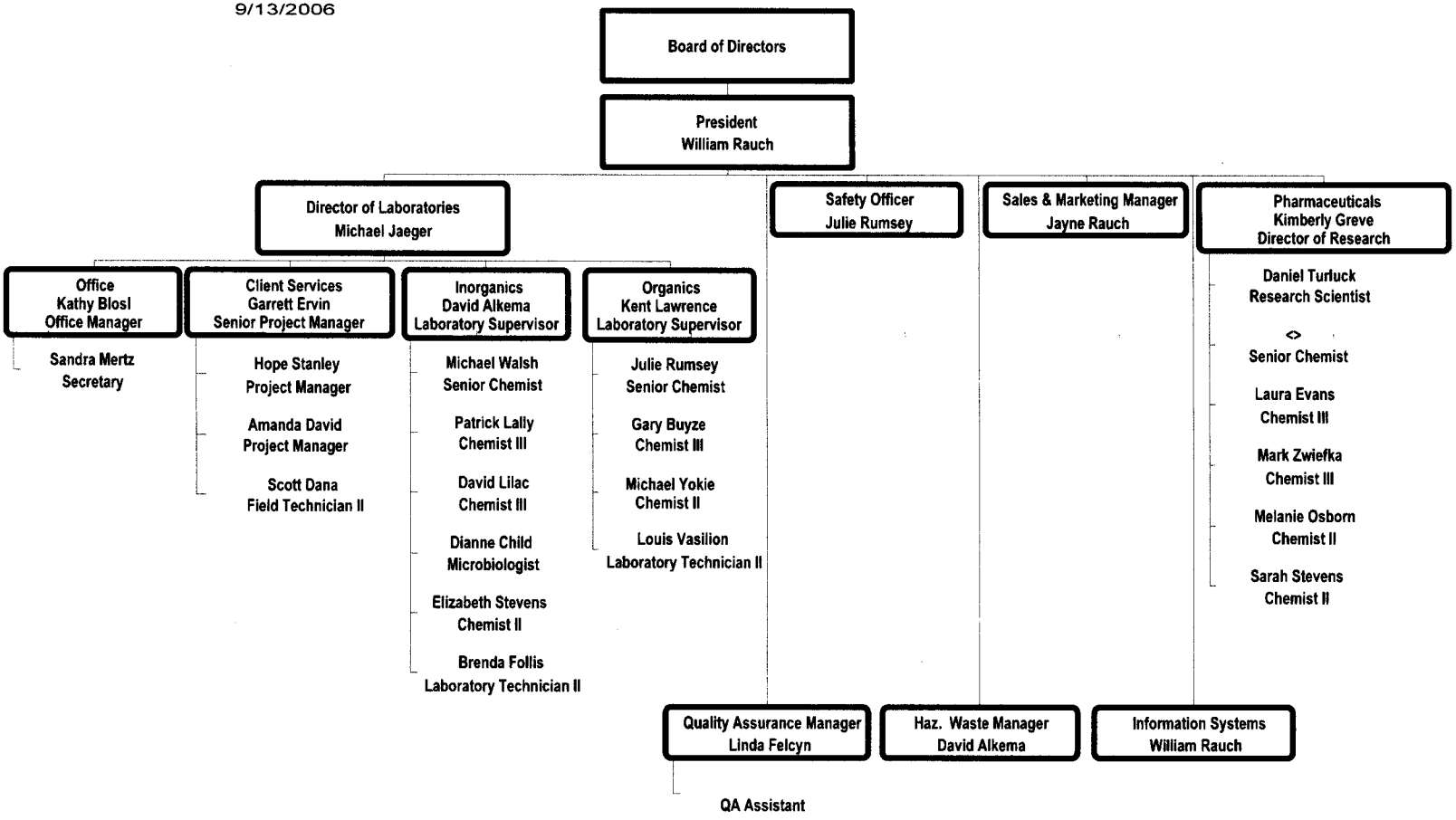
Quality Assurance Manager: Linda Felcyn _____ Date: Nov. 27 2006
Linda M. Felcyn

KAR Laboratories President: William G. Rauch _____ Date: 11-27-2006
William G. Rauch

KAR *Laboratories, Inc.*

ORGANIZATION CHART

9/13/2006



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2. SAMPLE COLLECTION

- 2.1 The Importance of Proper Sample Collection
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3. SAMPLE RECEIPT AND HANDLING WITHIN THE LABORATORY

- 3.1 Sample Receipt
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INTRODUCTION

1.1 Corporate Core Values

1.1.1 Clients

- Provide a wide range of services
- Keep promises, meet commitments
- Maintain high quality
- Charge reasonable fees
- Promote open & timely communication (client focus, client-driven)

1.1.2 Employees

- Promote employee involvement and empowerment
- Provide a generous, comprehensive benefit package
- Seek employee input, comments & criticisms
- Provide continuing education and training
- Encourage teamwork and cooperation
- Provide opportunity for advancement
- Recognize & acknowledge contributions of all employees

1.1.3 Ethics

- Treat all individuals with dignity and respect
- Ethical waste management: disposal, recycling, reuse, & minimization
- Be fair when billing for services
- Accept work only when we can provide something of value to our client
- Follow established SOPs and current approved methods
- Report complete and accurate data
- Pursue knowledge of environmental laws and regulations

1.1.4 Corporate Responsibility

- Act in a professional and responsible manner
- Support charitable causes and community programs

- Maintain a high level of involvement in professional associations
- Market, advertise, and promote KAR Labs appropriately
- Build relationships and reputation with Government officials/regulators
- Promote environmental awareness

1.1.5 Profits

- Make KAR a good investment for shareholders
- Provide substantial profit sharing to employees
- Provide stability and security for employees
- Minimize expenses wherever possible
- Increase efficiency/effectiveness
- Maximize revenue: expand client base, increase range of services offered

1.1.6 Quality

- Provide a high quality product (report) and high quality services
- Satisfy client requirements for timeliness
- Innovate: stay abreast of improvements in technology (leading edge)
- Provide a high level of service, client focus, and expertise
- Practice continuous improvement
- Adequately maintain building, laboratories, and grounds

1.2 Quality Control and Quality Assurance Programs

1.2.1 This manual is intended to be a general overview of the quality assurance program at KAR Laboratories, Inc. for the environmental testing area. Detailed, step-by-step procedures regarding Quality Control can be found in the appropriate Standard Operating Procedure (SOP). For example, the general requirements for sample receipt are outlined in this document, but the exact steps are found in the SOP.

1.2.2 **Definition of Laboratory Quality Assurance**: A program of techniques and procedures to ensure that the facilities, equipment, personnel, methods, practices, records, and controls are in compliance with regulations set forth by the Environmental Protection Agency (EPA).

1.2.3 **Definition of Laboratory Quality Control**: The techniques whereby the variables of the analytical processes are monitored and maintained within an established limit.

1.3 Quality Assurance Manager

It is the responsibility of the Quality Assurance Manager to design, implement, manage, and enforce the laboratory's overall Quality Assurance Program that ensures that laboratory data are produced and evaluated in compliance with quality standards. Assessments of the overall laboratory compliance are performed through periodic reviews of the facilities, systems, and processes. The Quality Assurance Manager maintains the Quality Assurance Manual and is responsible for its biennial review and update.

To ensure that QA Manager has adequate authority, the individual shall be a manager who has an independent reporting structure unrelated to laboratory managers and supervisors on the Organization Chart. The QA Manager is appointed by the president of the company.

1.4 Director of Laboratories

It is the responsibility of the Director of Laboratories to manage the overall operations of the environmental laboratory and supervise senior laboratory staff. The director ensures compliance with federal, state, and local regulations, corporate policies, and technical SOPs as well as to ensure that there is complete and accurate documentation on all projects. The director reviews and validates all laboratory results and signs the laboratory reports.

1.5 Laboratory Information Management System (LIMS)

The computerized LIMS system is at the center of storage, management and retrieval of KAR's QC information. The statistical nature and volume of QC data favors a computerized system. All information regarding samples and the associated quality information is immediately and easily available because every staff member has access to LIMS through a networked computer at their desk.

Every step of quality is tracked, and QC control charts are automatically generated and displayed during data entry. LIMS assists quality by providing:

- Printed sample labels
- Work lists that can be customized by the department supervisor
- Automatic uploading of instrumental data for Matrix Spikes and Matrix Spike Duplicates, calculations of Relative Percent Difference and Percent Recovery and comparison to method-specific limits
- Validation routines
- Report generation by several formats via hardcopy or electronic media
- Minimum and maximum values are assigned to data fields and format limited to mo/da/yr for consistency
- Extensive use of "Lookup" tables to provide exactly the same naming in Client information, test names, units, qualification codes and sample types and other areas as well
- Automatic data-entry validity checks for completeness (all of the following: concentration, units, date analyzed, analyst)
- Correct units for the sample type
- Denies impossible concentration for the units specified (example <0% or >100%)
- Rounding, concentration/dilution factoring and significant figure manipulations for instrumental data uploads
- Spike and Duplicate data must be within statistically-based QC limits for the matrix type with required "Reason for QC Failure" documentation upon acceptance of data which falls outside of QC limits
- Quality Control Charting is available for the parameter by pressing one key
- Automatic Validation Checks:
    - Missing units
    - Concentration outside the range for the units

- Concentration outside the likely range for the test
- Missing digit preceding a decimal point
- Holding time exceeded
- Missing comma separator on numbers >9999
- "Dissolved" concentrations greater than "Total"
- Unlikely relative BOD, COD and/or TOC relationships
- Hexavalent Chromium exceeding Total Chromium
- Printed copy of test results sorted via "test" (a proofreading aid)
- Printed copy of complete Ion Balance calculations with anion/cation listing, measured Solids vs. calculated Dissolved Solids agreement and measured Conductivity vs. calculated Conductivity agreement
- Printed copy of all client correspondence, additions, deletions and changes that relate to the project

1.6 Report Options

Upon completion of a project, KAR Labs issues a Standard Analytical Report which generally does not contain QA data. If QA data is desired, a "QA Analytical Report" should be requested (at no charge). This report contains spiked sample, duplicate sample, and analytical batch data with statistical calculations, instrument detection limits, sample analysis date, analyst, method used, and other data. All Quality requests and inquiries should be directed to the QA Manager.

If a client requires special or unique data quality objectives, those objectives must be addressed prior to the sampling event.

2. SAMPLE COLLECTION

2.1 Importance of Proper Sample Collection

Sampling is considered to be the single most important factor in an analytical scheme because all subsequent steps in data generation can only be a reflection of the sample that was submitted for analysis. For this reason, it is imperative that no analytical program be conducted without an adequate Sampling Plan that ensures

representative samples will be collected and that the integrity of those samples will not be compromised.

2.2 Elements of the Sampling Plan

A Sampling Plan must be designed by an individual who is familiar with both the various sampling devices and the analytical requirements. This familiarity is essential because (1) certain sampling devices are made of materials that may contaminate the sample, (2) cross contamination of samples may occur if the sampling device is not cleaned properly, (3) routine sampling methods may not be applicable to a sample that is to be analyzed for a unique parameter, and (4) the method of employing the sampling device may affect the integrity of the sample.

Whether the sampling responsibility lies with the client or KAR Laboratories, the following items must be in place to ensure the reliability of the data:

2.2.1 A properly designed Sampling Plan.

2.2.2 Sampling containers that have been properly cleaned and labeled for their intended use (see Appendix B for an example).

2.2.3 Containers that satisfy requirements for sample size which will likely include provisions for field duplicates and field blanks.

2.2.4 Containers that optimize conditions necessary for replication of results and preservation of the chemical integrity of the sample.

2.2.5 Chemical preservatives or refrigeration to satisfy the requirements of the analytical method and/or regulatory requirements (e.g., Appendix A).

2.2.6 Special provisions for project-specific data quality objectives.

3. SAMPLE RECEIPT AND HANDLING WITHIN THE LABORATORY

3.1 Sample Receipt

Sample integrity can be seriously compromised if samples are not preserved and stored in a proper and timely manner after they are received, as in Appendix A, or if samples are not analyzed in a timely manner because the record of time sampled and/or time of arrival has not been adequately documented.

It is the responsibility of the Client Services Department to completely record (input) all necessary information about a sample or sample set (KAR calls this a “Project”) into the computerized Laboratory Information Management System (LIMS). This must be done as soon as possible (typically the same day that the samples were received). Because all staff members have their own networked personal computer at their desk, the sample information is immediately available to the entire staff including the analyst responsible for the test. Management and clerical staff utilizes the LIMS to track work progress to ensure that no sample or test is overlooked.

Subsampling is often necessary because of varying preservation requirements. Such subdivisions are performed at sample login by a member of the sample login team to ensure the identity and integrity of sub-portions without deterioration or contamination.

Preservation, storage, and holding times are observed in accordance with EPA guidelines. These are automatically tracked by the Laboratory Information Management System (LIMS), and violations are automatically flagged on internal and final reports. Long-term storage of samples depends upon several factors which include the clients' request, expiration of holding time, hazardous properties, legal evidence (or possibility of legal evidence), and sample amount. Samples are always maintained for at least two weeks after the analytical report has been mailed or after a specified holding time has expired, whichever is longer.

3.2 Sample Handling

This procedure outlines the elements and steps involved in the handling of samples received for analysis at KAR Laboratories.

3.2.1 Samples are received directly from the client or from contracted carriers. Sample Receipts and Chain-of-Custody (see Appendix B for an example) documents are prepared at this time. Indelible, reproducible ink must be used for all recordings.

3.2.2 The project is activated by logging the project into the computerized Laboratory Information Management System (LIMS). The LIMS assigns a Project Number which is entered into the master projects data base. The established format must be followed by entering all requested information.

3.2.3 Each sample is assigned a unique identification number. This is a numerical or sometimes alphabetical suffix separated from the Project Number by a hyphen (example 891234-01). The assigned identification number shall be printed onto the LIMS-generated sample label or written onto the sample or sample container in ink or other permanent marker.

3.2.4 All pertinent information provided with the sample is then entered into the LIMS and is peer-reviewed for accuracy as a final check.

3.2.5 Samples are preserved and/or subsampled by Client Services. Samples are then routed to the proper laboratory for storage. The type of environment chosen for sample storage will be consistent with method protocol. All sample and subsample labels include a minimum of the KAR Laboratories Project Code # and the chemical preservative used, if any. Computer-generated labels are used whenever possible.

3.2.6 Analyses are carried out according to specified analytical procedures.

3.2.7 After all testing is complete, samples are returned to their respective storage areas and retained for a period of time consistent with approved holding times for the tests.

3.3 Chain-of-Custody

To ensure that the necessary chain-of-custody procedures are in place for the various requirements of its clients, KAR Laboratories implements one of several systems.

All samples received for analysis must use at least a KAR Laboratories Sample Receipt/Chain-of-Custody Record (an example in Appendix C). Some clients may prefer to use their own chain-of-custody form. This is considered supplemental to the KAR Chain-of-Custody Record.

KAR Laboratories has tamper-proof custody seals which can be used on sample containers. These custody seals are made of a special adhesive foil, and once installed at the bottle-cap interface, they cannot be removed without clearly indicating that the container was opened.

Upon request, a sample custodian may be assigned the responsibility of safeguarding the samples and ensuring that the chain-of-custody is maintained. To achieve this end, a locked storage receptacle accessible only to the sample custodian is used.

4. LABORATORY SUPPLIES, CHEMICALS AND EQUIPMENT

Glassware, chemicals, and equipment used at KAR Laboratories are selected to conform to the specifications defined in the method of analysis. Items not specifically mentioned in a particular method of analysis should conform to Good Laboratory Practices (GLPs). For example, Class A volumetric glassware is usually used for measurements that require a volume determination. If an item is available in a disposable configuration, then that option is sometimes preferred to avoid cross-contamination or introduction of other contaminants to a sample. Once it has been established that a particular supplier or manufacturer has a good record of supplying an item of suitable utility, ordering that item from a different source should be avoided.

4.1 Laboratory Glassware

4.1.1 **Cleaning Procedures**

4.1.1.1 General labware washing will include (at a minimum) a detergent wash in hot water, two rinses with ASTM Type II reagent water (also known at KAR as deionized [DI] water). The labware is then inverted and suspended to dry. An automated dishwashing system is available for routine glassware washing.

4.1.1.2 Labware for trace metals analysis includes a detergent wash in hot water, tap water rinses, one acid rinse followed by two rinses with ASTM Type II reagent water.

4.1.1.3 Purgeable hydrocarbon labware consists of cleaning as outlined in 4.1.1.1.

4.1.1.4 Pesticides and acid/base-neutral extractables labware incorporates the cleaning outlined in 4.1.1.1 followed by one rinse using pesticide-grade acetone and one rinse using pesticide-grade methylene chloride. The containers are then inverted and suspended to dry.

4.1.1.5 In all cases the sample bottle cleaning recommendations of the appropriate method and/or SOP should be followed.

4.1.2 **Testing for Contamination.** Contamination is monitored on a routine basis by the analysis of Laboratory Method Blanks (LMB). Reagent water is carried through all steps of an analysis parallel to the samples being analyzed. In this manner, any laboratory-borne contamination introduced by reagents, labware or the laboratory environment should be detected. A systematic troubleshooting scheme is implemented when an LMB fails.

The LMB is used throughout the laboratory. Most wet chemical methods include an LMB as part of the Standard Operating Procedure. Volatile organic instrumental analysis includes an LMB after every calibration standard, and as a means of verification that sample component carry-over from a previous high-level sample has not occurred.

4.1.3 **Storage.** KAR Laboratories has adequate storage capability and, therefore, has greater flexibility in providing an appropriate environment for special applications. Labware for everyday use is stored at a location isolated from potential contaminants. Specially cleaned labware such as those designated for the Organic Laboratory are stored separately in the Organic Preparation Lab in a covered enclosure. Labware designated for trace metals analysis are stored in the Trace Metals Laboratory in a covered cabinet to avoid potential dust contamination. Light, heat, and/or moisture sensitive items are stored in a special room intended for this use. Other items not demanding any special storage considerations are stored to provide the most efficient retrieval of the item.

4.2 Chemicals and Reagents

4.2.1 **Purity Specifications -** All chemical reagents and gases are selected to meet or exceed the specifications defined in the method of analysis.

4.2.2 **Receipt procedures -** All chemicals, reagents, gases, and other expendables are labeled upon receipt with the date received. Upon opening, the date is recorded on the label. An expiration date is assigned to each chemical/reagent based on the manufacturer's recommendation or on KAR Laboratories' dating practices.

4.2.3 **Shelf Life -** Most reagents/chemicals have a limited shelf life. Purchased chemicals are labeled with the date received, the date it was opened, the expiration date, and the storage conditions. Reagents are labeled with the date it was prepared, the name or initials of the analyst who prepared it, the storage conditions, and the expiration date. Some laboratory Standard Operating Procedures require that associated reagents be prepared fresh daily at the time the assay is being performed. Other reagents such as prepared standard solutions and calibration compounds for atomic absorption and gas chromatography are replaced on an expiration basis. In no case may a calibration standard be used after one year has elapsed. The appropriate SOP or method guidelines must always be followed.

4.2.4 **Laboratory Reagent Water -** ASTM Type II reagent water (also called RO water, DI water, or D-H_2O) is supplied by an on-site Reverse Osmosis system with an on-demand high capacity storage tank. This system is centrally plumbed to provide reagent water to all laboratories. Reagent water purity (conductivity) is continually monitored and recorded daily. Monthly, all common parameters including organics, trace metals, general water quality, and microbiology are analyzed. If the concentration of any contaminant approaches the laboratory's reporting limits, reporting of that parameter to external clients must be suspended until the problem is corrected.

4.2.5 **Documentation -** To document traceability of chemicals and reagents, the manufacturer and lot number of the item used is recorded as part of the laboratory assay preparation.

4.3 Equipment/Instrument Calibration Checks

4.3.1 All repeating pipettes are calibrated weekly.

4.3.2 Balances are checked daily and calibrated quarterly by in-house staff with certified Class S weights (see examples in Appendices E and F). All completed calibration sheets are reviewed and available for inspection. In addition, balances are calibrated annually by an external contractor.

4.3.3 Analytical instrumentation's calibration is checked through the use of surrogates, spikes, blanks and calibration standards.

4.3.4 The temperature of all sample refrigerators, freezers, and BOD incubators is checked daily. Microbiology incubators are checked twice daily. All completed Log Sheets are forwarded to QA and are available for inspection (see an example in Appendix D). Thermometers and thermocouples are checked annually against an NIST-traceable source.

5. ANALYTICAL METHODOLOGY

5.1 Analytical Methods

The most current sources for analytical procedures are used as references to develop KAR Laboratories' analytical SOPs. The SOPs are controlled and followed throughout the laboratories and are based on methods from:

5.1.1 Standard Methods for the Examination of Water and Wastewater, American Public Health Association, American Water Works Association, Water Environment Federation.

5.1.2 Methods for Chemical Analysis of Water and Wastes, U.S.EPA.

5.1.3 Annual Book of ASTM Standards, American Society for Testing and Materials.

5.1.4 Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW-846, U.S.EPA.

5.1.5 Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, U.S.EPA.

5.2 Standard Operating Procedures (SOP)

KAR Laboratories maintains SOP manuals which contain analytical SOPs of test procedures routinely performed in each laboratory and appropriate general procedures (an example of the SOP list is located in Appendix G). Analytical SOPs supercede general SOPs. All new SOPs or revisions to existing SOPs are approved, tracked and controlled by QA. The following format, where appropriate, is suggested when preparing an SOP:

5.2.1 **Title/Header -** The property, analyte, or class of compounds being measured. Also includes the SOP number, date issued and next required review date.

5.2.2 **References -** A published reference(s), usually regulatory approved, which defines the basic analytical methodology. Other KAR Laboratory SOPs may be cross referenced.

5.2.3 **Application -** Types of sample matrices with which the method may be used or a brief description of the SOP's purpose and scope.

5.2.4 **Discussion -** Information about the procedure or background information that aids the analyst in understanding the purpose of the method.

5.2.5 **Summary of Method -** A brief description of the procedural elements of the method.

5.2.6 **Sample Handling -** Any specific concerns with samples or reagents to alert the analyst of potential problems.

5.2.7 **Interferences -** A general overview of matrices or conditions that have the potential for interfering with an accurate measurement. Also includes any steps that will compensate for the interference.

5.2.8 **Apparatus -** A list of equipment/instrumentation (manufacturer and model) and labware including special conditioning, class or required assembly.

5.2.9 **Chemicals and Reagents -** Chemicals that are specific for the test are summarized, indicating the required purity. This section may also include instructions on reagent preparation, shelf life, standardization, and/or disposal, if applicable.

5.2.10 **Safety -** Includes safety considerations that are unique to the method.

5.2.11 **Preparation of Standards.** This section details the proper method for making all standards and quality controls used in the analysis.

5.2.12 **Procedure -** A detailed description of each step that is to be taken in the laboratory.

5.2.13 **Calculations -** The mathematical steps used to complete the analysis. Includes a sample calculation, if it will be helpful, and specifies the number of significant figures to report. May include a blank analytical bench form, if it will help explain any calculations.

5.2.14 **QA/QC -** A list of additional quality assurance and quality control functions specific to the SOP which may include the following:

5.2.14.1 QA procedures specific to the method, especially equipment and reagent checks that should be performed routinely

5.2.14.2 QC parameters specific to the method: Batch size, reference materials, data handling, etc.

5.2.14.3 Quantitation Limit - The concentration below which all results are reported as "less than," based upon the written procedure which specifies the sample size and any dilution or concentration inherent to the test.

5.2.15 **Data Reporting/Electronic Systems Interface.** A detailed description of what to report, significant figures, units, and use of LIMS to facilitate reporting.

5.3 Auditing Performance

Method performance is checked in the following ways:

5.3.1 KAR uses two standard deviations from the mean (95% confidence) for warning limits and three standard deviations from the mean (99% confidence) for control limits.

If the maximum allowable range is set by regulation, then the statistical range may not exceed the regulated maximum.

When statistical data is unavailable then $\pm$ 20% can be used (although this may be parameter specific) unless a regulation exists that is more restrictive.

Values which are out of control will be investigated. Data that has failed QC limits (out of control) will be qualified (flagged as failing QC) on the final report to the client.

5.3.2 Duplicate analyses are performed at least on one in every ten samples. If the difference between duplicate sample results exceeds that specified in the SOP, then the cause must be determined and the samples may have to be reanalyzed. Duplicate measurements are automatically tracked and charted by LIMS.

5.3.3 Spiked samples are analyzed one in every ten samples. If the recovery falls outside the range specified in the SOP, then the cause must be determined and the sample reanalyzed if necessary. Spike measurements are automatically tracked and charted by LIMS.

5.3.4 KAR Laboratories subscribes to a commercially available QC check sample program for wastewater and Priority Pollutants on a quarterly basis.

5.3.5 Performance Evaluation Samples (also called proficiency testing samples) are analyzed and the results are submitted to either the EPA (for certain drinking water parameters on a quarterly basis) or to other oversight bodies.

5.3.6 EPA QC Check Samples are analyzed on a routine basis.

5.3.7 Split samples with other labs and regulatory agencies are analyzed.

5.3.8 Surrogate compounds are used routinely in organic analyses to monitor recovery.

5.3.9 Internal audits and inspections are performed periodically by the QA department to build laboratory strengths and to detect and eliminate deficiencies.

5.4 Method Validation

In most cases method and/or operator verification/validation will be established by the successful completion of a Method Detection Limit Study (MDL). This study establishes the accuracy, precision and minimum detectable level on reagent water. This is true for EPA methods and Standard Methods which comprise the vast majority of methods used at KAR Laboratories.

In cases where a written validated method is unavailable, whatever method development or modification is undertaken shall be done so in a context that concludes with at least an informal method validation study. Most commonly this will involve a combination of spiked samples, replicate samples, linearity study, and confirmation of positive results by at least one other method or technique.

6. INSTRUMENT PERFORMANCE

6.1 Sensitivity, Specificity, and Stability

The acceptable performance of an instrument with respect to sensitivity, accuracy, and precision is demonstrated before the analyzing samples.

Some sample matrices will not allow a low Limit of Detection to be achieved. Samples of this type are noted as such on the analytical report along with the estimated Limit of Detection. In all cases sensitivity must be maintained to meet regulatory requirements. All acceptable cleanup procedures should be employed when necessary. Unnecessary or arbitrary sample dilutions are not allowed.

6.2 Monitoring Performance

6.2.1 **Monitoring Performance for General Parameters.** Instrument performance is monitored in several ways. Problems may appear during the daily

calibration. When such problems arise, it will become apparent whether the instrument can achieve the necessary performance. Quality control check samples are used frequently. Blanks monitor for contamination. Replicate samples and spike samples are used at a frequency of approximately one in ten samples. Any QC sample that is out of control according to the control chart must be investigated and documented. If, after investigation, the measurement continues to be out of control, the data must be qualified accordingly on the final report. If the statistical QC acceptance criteria for relative percent difference (RPD) between duplicate samples is less than 5%, then a value of 5 will be used for acceptance.

6.2.2 **Monitoring Performance for Trace Metals**

Parameters	ICV	CCV	LCS	ICB/ CCB	Dup. RPD	Spike Recovery
Furnace	90-110%	85-115%	85-115%	<RL	20%	80-120%
ICP and Flame	DW 90-110% Others 90-110%	90-110%	85-115%	<RL	20%	75-125%
Mercury	DW 95-105% Others 90-110%	DW 90-110% Others 80-120%	85-115%	<RL	20%	aq 75-125% non 70-130%

QC Requirements

- Correlation coefficient must be $\geq$ 0.995
- ICV (from different source) immediately after calibration must be within 90-110% of true value
- ICB immediately after ICV must be < RL
- CCV and CCB after every 10 analyses
- One matrix spike and one duplicate analysis every 10 samples
- One LMB and one LCS for every preparation batch
- Spike recoveries and duplicate RPD must meet established performance limits

- ICP/MS must have acceptable internal standard recovery

Codes:

- DW Drinking Water
- ICV Initial Calibration Verification
- ICB Initial Calibration Blank
- CCV Continuing Calibration Verification
- CCB Continuing Calibration Blank
- LMB Laboratory Method Blank
- LCS Laboratory Control Standard
- RPD Relative Percent Difference
- RL Reporting Limit
- ICP/MS Inductively Coupled Plasma/Mass Spectroscopy

Notes:

- Data is invalidated by unacceptable ICV, ICB, CCV, CCB, or IS results.
- Unacceptable spike, duplicate, LMB, or LCS results are investigated before validating data.
- Spike and duplicate limits are generally based on the method's observed performance with real samples.

6.2.3 **Monitoring Performance for Gas Chromatography.** Method performance requirements are written into the SOP. In most cases this will result in one matrix spike/matrix spike duplicate analysis for each analytical batch.

6.3 Calibration

Instruments are calibrated prior to each day's use in accordance with the method used or the SOP and repeated periodically throughout the day as required. The usual procedure is first to establish a linearity graph consisting of a blank and at least three standards. This establishes the linear working range for the instrument. When the linear range has been established and documented, the calibration is

checked daily with at least two points, a blank and a standard. If the results of this daily calibration check demonstrate agreement with the established calibration curve, then it may not be necessary to run additional standards. These calibration procedures will vary from method to method and in all cases the appropriate Standard Operating Procedure should be consulted.

6.4 Maintenance

Instruments are installed and maintained to maximize their usefulness and to minimize downtime. Every analytical instrument system has a log book to record maintenance procedures, recurring or intermittent problems which may not allow immediate repair, and troubleshooting incidents. All factors that may affect the system are to be recorded. Each entry is dated and signed, and any suggestions or recommendations are also included. All balances are checked and serviced annually by an outside contractor.

7. DATA HANDLING AND RECORD KEEPING

Record keeping is essential to all laboratory activities. Only by properly maintaining permanent records using indelible ink and contemporaneous documentation can it be determined that the analytical data generated is reliable. If appropriate records are not made, final results may not be useable.

7.1 What Records to Keep and How Long

It is the responsibility of each individual employed by KAR Laboratories to maintain complete records of their work in the detail and format prescribed by the department supervisor and/or SOP. The minimum requirement shall be that all experimental work be recorded at the time performed and be described in sufficient detail to make it understandable to one familiar with analytical chemistry yet not directly connected with the work. All such records (termed raw data) will be retained for a minimum of seven years, then destroyed. All records associated with quality control systems

(e.g., SOPs, audit reports, training files) will be retained at KAR Laboratories for a minimum of seven years.

7.2 Protocol and Forms

All experimental data will be recorded in indelible ink, usually in a bound laboratory notebook. The name of the analyst shall be clearly indicated on all work so that there is no question who is responsible. All numerical data shall comply with the following format: (1) a digit must always precede a decimal point, and (2) a comma separator shall be used for all numbers five digits or larger.

Entry errors, deletions, or corrections shall be indicated as such by drawing one single line through the text, table, drawing, or graph that is to be canceled. These errors are further notated by the date and initials of the person responsible for the changes. Erasures and use of correction fluid is not permitted. Transcriptions shall be avoided whenever possible.

All laboratory record books are the property of KAR Laboratories and may not be removed from the premises without proper authorization. Individuals who wish to maintain other records may do so, after supervisory approval, but records shall be in addition to the complete and permanent records mentioned above. Any notation intended to indicate the same value (such as “ditto” marks, arrows, etc.) is acceptable except for recording direct measurements (e.g., sample weights). Each direct measurement is individually recorded contemporaneously; that is, recorded at the time the measurement is performed.

Data in the form of charts, instrument recordings, and printouts will be given suitable identification and maintained in a manner similar to written records.

All analytical results, when entered into the LIMS system, shall include the date analyzed and the analyst's name or initials. Any other information relevant to the test should be included in the comments field.

All LIMS data is hardcopied and filed on a project basis. All electronic data is backed-up daily onto magnetic tape and kept for seven years, then destroyed.

7.3 Important Aspects of Documentation and Qualifying Data

In view of the possible legal implications that accompany the generation of analytical data, all data shall be recorded in such a way as to preclude its discrediting at any time. Any deviation from the method protocol that may affect the results should be reported on the analysis report. This includes such items as expired holding times or improperly collected samples.

7.4 Reporting Results

7.4.1 **Significant Figures.** KAR Laboratories uses "significant figures" when reporting results. This means that all digits in a reported result are expected to be known definitely except for the last digit which may be in doubt. If more than a single doubtful digit is carried, then the extra digits are not significant and, therefore, should not be reported (see section 7.4.2). Analysts shall report only such figures that are justified by the accuracy of the work. Rounding and significant figures apply only to the final, reported results. Intermediate calculations necessary to arrive at a final result shall not be rounded.

7.4.2 **Rounding.** Digits that are not significant will not be reported. To round a number to the reported value, the digits 6, 7, 8, or 9 that follow the significant digit will round the reported value up by one unit. If the digit 0, 1, 2, 3, or 4 follows the significant value, the reported value is not altered. If the digit 5 (and no other additional digits are included) follows the significant value, the reported value is rounded to the nearest even number (e.g. 114.5 rounds to 114 and 115.5 rounds to 116). If the digit 5 has additional digits following it that are non-zero, the reported value should be rounded up. Exceptions to

this rule may be necessary depending on software that accompanies analytical instrumentation or used for report generation.

7.4.3 **Format.** Values which contain a decimal point shall always be preceded by an integer or zero (e.g., 0.31). Units shall always accompany a result except in cases where units do not apply.

7.4.4 **Detection Limits.** A "less than" sign (<) will precede the detection or quantitation limit in cases where the analyte is not detected or quantitated. Calculated values less than these limits shall be stated as such without rounding. For example, a calculated final results of 0.98 for a test that has a detection limit of 1 shall be reported as "<1" and not rounded up to 1.

8. DATA INTEGRITY, CONFIDENTIALITY, CHALLENGES, AND DATA CORRECTION

8.1 Data Integrity

Data integrity is established through the use of data review, assay training, pre-employment agreements, secure data storage, and the backing up of electronic records. Data is either generated from validated systems or goes through review processes to ensure that reported tests reflect laboratory results. All QC results in support of tests are reviewed to make sure they are within established acceptance criteria and that all required quality control samples were analyzed. Each analyst has been shown to be proficient in their assigned assay (see Section 9.1) and have documented training on policies and procedures that support the laboratory. Data is stored in secure locations with limited access. Electronic data, after capture, is protected against erasure and tampering, as well as backed-up and stored off-site per internal standard operating procedures.

8.2 Confidentiality Agreement

Each employee is required to sign an agreement that ensures that all data and client information is kept confidential. Only laboratory supervision or higher are permitted to discuss laboratory results or client-specific information outside of KAR Laboratories unless otherwise instructed. Clients may view their sample results through the internet. This access is granted only through approval by KAR management. Clients are issued passwords and specific "rights" that allow only client-owned data to be viewed.

Terms of employment regarding non-disclosure agreements are further discussed in the KAR Employee Manual. Any employee or ex-employee who discloses trade secrets or confidential business information will be subject to disciplinary action. All outside jobs held by employees must be reported to KAR Laboratories' President in advance for approval. These restrictions avoid any activities that could diminish employee competence, impartiality, and integrity.

8.3 Data Challenges and Auditing Data Validity

End-users (clients) of laboratory data are encouraged to challenge any data that does not fit their expectations by requesting a data review from Quality Assurance or the Project Manager. All information shall be recorded on the KAR **QC Review Form** (see Appendix I for an example). Similarly, laboratory staff who suspect that data may be incorrect or inaccurate for any reason must immediately report the incident. The request will then be routed directly to the supervisor. In the event that the supervisor is unavailable and the review cannot wait, then the group leader of the area is consulted. The supervisor will then be notified of the request via e-mail or other means, but in no case should the supervisor go unaware of a data change/inquiry request. This is necessary to allow the supervisor to monitor system and analyst performance and ensure that corrective actions are taken.

8.4 Data Correction and Notification Procedures

Quality Assurance will be notified of all report revisions containing data changes. It

is the laboratory's responsibility to contact the client immediately of an error or, in the case of a review initiated by the client, within eight working hours. The telephone call is then followed up with a printed Revised Report mailed to the client if a review uncovers a reporting error. If a systematic or other long-term condition is found which could affect data quality and/or accuracy, an attempt must be made to ensure that all reported data that might be affected be handled in the manner specified above.

8.5 Laboratory Corrective Action

Each laboratory supervisor is responsible for the initiation of all corrective action processes performed within their section. KAR Quality Assurance (QA) is responsible for the coordination/oversight and/or documentation of all such activity. Documentation includes the nature of the problem, the name and affiliation of any person contacted regarding the problem, and the action taken to correct the problem. QA reviews the details regarding the outcome of any corrective action/problem resolution and initiates any procedural changes deemed necessary.

In the case of instrument failure or procedural problems, the analyst is responsible for terminating the analytical run and contacting the laboratory supervisor. The problem may be evaluated through consultation with QA and the appropriate corrective action taken. Analyses will resume when proper operation of the instrument is confirmed.

As part of the data evaluation process, the Laboratory Director or QA will initiate corrective actions when reported results exceed acceptance limits or when the QC requirements for a given analysis are not satisfied.

When the deviation is known to have a definite effect on data quality, QA will automatically reject the data. The appropriate laboratory personnel are notified. In some cases, QA may choose to confer with the client's Project Manager to help determine the effect of the deviation on data usage. The Project Manager notifies

the appropriate personnel of the situation. A decision on the corrective action is made and submitted to KAR's QA along with the necessary steps to be taken. KAR's QA qualifies or rejects the affected data. Data qualified as approximate will be reported as such, and the deviation will be described in the report.

9. PERSONNEL

9.1 Training

The training for specific tasks is performed under the guidance of a laboratory supervisor or an experienced analyst in conjunction with written Standard Operating Procedures. In all cases, an analyst must first demonstrate competency for any assigned test by successfully meeting precision, accuracy, and detection limit requirements defined in the SOP or the Method. Appropriate method detection limits (MDLs) are entered into the database and tracked by LIMS, and documented using the Initial Demonstration of Capability (IDC) Form (see examples in Appendix J & K) which is placed in the individual's training folder located in the quality assurance office. Employees (temporary or otherwise) without formal laboratory training and/or experience will not be assigned any analytical tasks unless approved by Quality Assurance. This includes (but is not limited to) measuring samples, labeling samples, and filtering.

Formal educational backgrounds for technicians include a minimum of an associates degree or equivalent. Chemists and microbiologists require a four year science degree or three years of experience. Continuing education is encouraged for all employees, the expense of which is covered by KAR Laboratories according to current company policy.

9.2 Safety

KAR Laboratories maintains an active safety program which includes a designated Safety Officer. The facility operates in a manner that is safe for both the employees

and the environment. Protective equipment is worn and wastes generated in the lab are disposed in a safe and environmentally sound manner. All chemicals are appropriately labeled and Material Safety Data Sheets are maintained on the property, easily accessible to all employees.

Employees are instructed to avoid working in the laboratory with hazardous chemicals or processes unless someone else is on the premises. This includes procedures where personnel are handling large glassware, flammable or explosive substances, chemically hazardous samples, or large amounts of solvents, acids, bases, or strong oxidizers.

Any time an employee is in a situation that requires the use of protective gloves, the selected glove must be appropriate to guard against the possible exposure in the laboratory. Several types of gloves are available which are specifically targeted to prevent exposure to certain chemical, biological, or environmental contact. After using any type of glove, all analysts are instructed to thoroughly wash their hands.

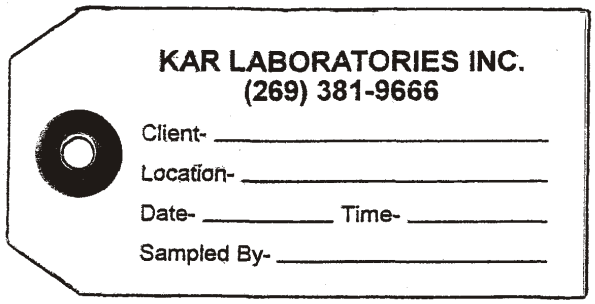
Laboratory surfaces that have the potential to be exposed to any type of biomatrix is decontaminated either after the work is finished or before the analyst leaves the area. Decontamination is achieved by either exposing the surface to a 10% chlorine bleach solution (prepared within 24 hours) or a dilute Vesphene solution. The surface must be in contact with the decontamination solution for at least 10 minutes.

CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR AQUEOUS MATRICES[A]

Parameter	**Container**[1]	**Preservation**	**Maximum holding time**
Inorganic Tests:			
Chloride	P, G	None required	28 days
Chlorine, residual	G	Analyze immediately	0.5 hours
Cyanide, total and amenable to chlorination	P, G	Cool, 4ºC; if oxidizing agents present add 5 mL 0.1N $NaAsO_2$ per L; adjust pH>12 with NaOH.	14 days
Hydrogen ion (pH)	P, G	None required	24 hours
Nitrate	P, G	Cool, 4ºC	48 hours
Sulfate	P, G	Cool, 4ºC	28 days
Sulfide	P, G	Cool, 4ºC, add zinc acetate and NaOH	7 days
Metals:			
Chromium VI	P, G	Cool, 4ºC	48 hours (target 24 hrs)
Mercury	P, G	HNO_3 to pH<2	28 days
Metals, except chromium VI and mercury	P, G	HNO_3 to pH<2	6 months
Microbiological Tests:			
Coliform, total	P (sterile)	Cool, 4ºC	30 hours
Colilert (presence/absence)	P (sterile)	Cool, 4ºC, 0.008% $Na_2S_2O_3$	30 hours
Other microbiological tests			Generally 24 hours
Organic Tests:			
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4ºC, HCl	14 days
Benzidines	G, Teflon-lined cap	Cool, 4^{O}C, store in dark	7 days until extraction, 40 days after extraction
Chlorinated hydrocarbons	G, Teflon-lined cap	Cool, 4ºC	7 days until extraction, 40 days after extraction
Haloethers	G, Teflon-lined cap	Cool, 4ºC	7 days until extraction, 40 days after extraction
Nitroaromatics and cyclic ketones	G, Teflon-lined cap	Cool, 4ºC, store in dark	7 days until extraction, 40 days after extraction
Nitrosamines	G, Teflon-lined cap	Cool, 4ºC, store in dark	7 days until extraction, 40 days after extraction
Oil and grease	G	Cool, 4ºC, H_2SO_4 to pH<2[2]	28 days
Organic carbon, total (TOC)	P, G	Cool, 4ºC, H_2SO_4 to pH<2[2]	28 days
PCBs	G, Teflon-lined cap	Cool, 4ºC	7 days until extraction, 40 days after extraction
Pesticides	G, Teflon-lined cap	Cool, 4ºC	7 days until extraction, 40 days after extraction
Phenols	G, Teflon-lined cap	Cool, 4ºC	7 days until extraction, 40 days after extraction
Phthalate esters	G, Teflon-lined cap	Cool, 4ºC	7 days until extraction, 40 days after extraction
Polynuclear aromatic hydrocarbons	G, Teflon-lined cap	Cool, 4ºC, store in dark	7 days until extraction, 40 days after extraction
Purgeable aromatic hydrocarbons	G, Teflon-lined septum	Cool, 4ºC, HCl to pH<2[3]	14 days
Purgeable halocarbons	G, Teflon-lined septum	Cool, 4ºC, HCl to pH<2[3]	14 days

[A]Table excerpted, in part, from Table II, 49 FR 209, October 26, 1984, p 28 and Standard Methods 18th edition
[1]Polyethylene (P) or Glass (G)
[2]KAR's preferred preservative is listed. Method allows use of either H_2SO_4, HCL or solid $NaHSO_4$.
[3]Free chlorine must be removed prior to addition of HCl by the appropriate addition of ascorbic acid.

APPENDIX A



KAR LABORATORIES INC.
(269) 381-9666

Client- _____

Location- _____

Date- _____ Time- _____

Sampled By- _____

KAR LABORATORIES, INC.

4425 Manchester Road

Kalamazoo, MI 49001 • (269) 381-9666

SITE NAME	DATE / INITIAL
LOCATION	TIME
ANALYSIS	PRESERVATIVE

PLEASE LEAVE THIS SPACE BLANK

KAR LABORATORIES, INC.

4425 Manchester Road

Kalamazoo, MI 49001 • (269) 381-9666

SITE NAME	DATE / INITIAL
LOCATION	TIME
ANALYSIS	PRESERVATIVE
KAR NO.	

Kar Labs 4425 Manchester Kalamazoo MI 49002	SITE NAME	DATE/INITIAL
	LOCATION	TIME
	KAR NO.	ANALYSIS

Appendix B

Client:	Project Name:	Requested Analyses	**KAR use only**
Attn:	Project #		Proj#:_____ Login:_____ Date: _____
Phone: ( ) Fax: ( )	P.O. #		c-of-c Y N labels Y N LODs Y N
Cell : ( ) Email:	Sampled by: Initials ☐ KAR ☐ Client __ __ __		contain Y N headspace Y N amount Y N hold time Y N rush cost Y N
Turnaround Time: ☐ 10-business days ☐ Emergency (quote) ☐ 5-business days (x 1.5) ☐ 3-business days (x 2.0) ☐ Monthly	Samples rec'd Cold: Y N Intact: Y N		

#	Sample Identification	Sample Info Date	Time	Matrix	Sample Containers Type	Size	#	Remarks

Relinquished By:	Received By:	Date/Time:	Notes:
Relinquished By:	Received By:	Date/Time:	

Appendix C

REFRIGERATOR DAILY TEMPERATURE LOG

Refrigerators are to be checked first thing in the morning. Acceptable Range is **1, 2, 3 or 4°C**. All corrective actions MUST be recorded in "Comments" column, use back side if needed. Submit to QA Dept. immediately upon form completion.

Date	Time	Init.	LI2	LI3	WIC	BA1	BA3	WQ1	V1	V2	SV1	SV2	SV3	SV4	SV5	Comments

DO Door was recently Opened
TA Temperature Setting was Adjusted
TB Thermometer Broken
TR Thermometer Replaced

RB Refrigerator Broken
RR Refrigerator Repaired
RD Refrigerator Defrosted

Reviewed by: _____
Date: _____

APPENDIX D

SOP: KP018

DAILY CALIBRATION LOG

Balance name_____S/N_____

Class S Weights S/N _____

Location: _____

Manufacturer range (_____x_____)g

IF CHECK FAILS

CONTACT QA IMMEDIATELY

OPERATING RANGE

Daily Check Weight

DAILY CALIBRATION

DATE	ANALYST	MASS(g)	OBSERVED	LIMITS	PASS	FAIL	ACTIONS/ COMMENTS
				+/- 0.-----			
				+/- 0.-----			
				+/- 0.-----			
				+/- 0.-----			
				+/- 0.-----			
				+/- 0.-----			
				+/- 0.-----			
				+/- 0.-----			
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				+/- 0.-----			
				+/- 0.-----			
				+/- 0.-----			
				+/- 0.-----			
				+/- 0.-----			
				+/- 0.-----			

REVIEWED DATE_____

APPENDIX E

SOP: KP018

QUARTERLY BALANCE CALIBRATION LOG

BALANCE NAME _____ S/N _____

External calibration mass (_____g) Class S Weights S/N _____

Location: _____

Manufacturer Range (_____X _____)g

OPERATING RANGE (____ X _____)g

BALANCE LEVELED

BALANCE CLEANED

LINEARITY CHECK

MASS	**OBSERVED MASS (g)**	**LIMIT**
	_____	**+/- 0.0-----g**
	_____	**+/- 0.0-----g**
	_____	**+/- 0.0-----g**
	_____	**+/- 0.0-----g**

MEETS CRITERIA

FAILS CRITERIA **Contact QA Immediately**

REPRODUCIBILITY CHECK

MASS USED _____G **MASS (g)**

_____

_____

_____

_____

_____

_____

_____

_____

CALCULATIONS

MEAN_____	**LIMIT**
STD DEV_____	**+/- 0.0-----g**
%RSD_____	**+/- 0.0-----**

MEETS CRITERIA

FAILS CRITERIA **Contact QA Immediately**

ACTIONS/COMMENTS:_____

_____

_____

ANALYST_____ DATE_____

REVIEWED BY_____ DATE_____

*Refererence:

APPENDIX F

APPENDIX G

KAR LABORATORIES ENVIRONMENTAL

STANDARD OPERATING PROCEDURE (SOP) INDEX

General/QA

KG021	Assignment, Review and Archiving of Laboratory Notebooks and Logbooks
KG022	Environmental Internal Audit/Inspection Program
KG023	Generation and Maintenance of Environmental SOPs
KG024	Environmental Laboratory Training Program
KG032	Environmental Data Reduction, Validation and Reporting
KG033	Faxing Completed Laboratory Reports
KG034	Chemical Inventory Procedures
KG035	Environmental Quality Control Charting
KG036	General Maintenance/Repairs and Preventive Maintenance for Environmental Laboratories
KG040	Storage, Archiving, and Disposal of Environmental Laboratory Records
KG066	Handling, Labeling, and Assigning Expiration Dates to Chemicals and Consumables
KG990	Electronic Data Deliverables
KGSDWA	Special Considerations for Drinking Water Samples
KP018	Calibration of Analytical and Top-Loading Balances
KP032	Facilities and Utilities: Use and Maintenance
KP033	External Audits
KP035	Freezer and Refrigerator Temperature Monitoring and Maintenance
KP036	Responsibility of Quality Assurance/Quality Control
KP044	Software Validation
KP900	Responding to Power Outages and Security Violations
KP908	Use and Maintenance of Supporting Laboratory Equipment

Client Services (Sample Receipt/Handling)

KC020	Sample Receipt, Handling and Storage (Chain-of-Custody)
KC030	New Project Login
KG031	Receiving and Reporting of Domestic Drinking Water Samples
KC100	Dissolved Sample Preparation of Metals Analysis
KC120	The Collection of Residential Water Samples for the Analysis of Bacteria
KC130	Collection of Industrial Waste Water Samples
KC200	Sampling for Ultra-Trace Mercury

KC220	Determination of the Conductivity of a Water Sample at 25° C
KC420	Measurement of pH in Water by the Electrometric Method

Microbiology

KB010	Operation of the Brinkmann/Tuttnauer 2540E Autoclave
KB030	Heterotrophic Plate Count by Pour Plate Method
KB035	The Detection and Enumeration of *Pseudomonas aeruginosa* Bacteria Using a Membrane Filter Technique
KB050	Monitoring Requirements and Equipment Maintenance for Microbiology Procedures
KB057	The Preparation of Media for Microbiological Analyses
KB060	Preparation of Phosphate Buffered Rinse/Dilution Water
KB064	Use and Care of the Water Baths in the Microbiology Lab
KB065	The Characterization of Gram-Negative Bacteria Using the API 20E Test Strip
KB070	The Preparation of a Positive Control for Coliform Analyses
KB100	Total Coliform Bacteria by the Membrane Filter Procedure
KB110	Total Coliform Bacteria by the ONPG-MUG (Colilert®) Procedure
KB150	The Detection and Semi-Quantitation of Total Coliform Bacteria using the Multiple Tube Fermentation Technique
KB155	The Detection and Semi-Quantitation of Fecal Coliform Bacteria using the Multiple Tube Fermentation Technique
KB200	Fecal Coliform Bacteria by the Membrane Filter Procedure
KB210	*E. coli* Bacteria by the Membrane Filter Procedure
KB300	The Presence - Absence Detection of *Salmonella* Bacteria
KB600	The Detection and Quantitation of Yeast and Mold in Water

Trace Metals

KM010	Glassware Washing - Trace Metals Laboratory
KM100	Mercury Analysis by Cold-Vapor Generation
KM120	The Preparation of Soil Fractions for Lead Analysis
KM150	Trace Metals Analysis Using the Perkin Elmer 4300 DV Inductively Coupled Plasma-Optical Emission Spectrometer
KM175	Acid Digestion of Sediments, Sludges, and Soils for Metals Analysis
KM200.8	EPA Method 200.8
KM200.8CAL	Calibration of Hewlett-Packard Inductively Coupled Plasma Mass Spectrometer
KM301	Aqueous Sample Preparation by Acid Digestion for Trace Metals Analysis using the Environmental Express Block Digester

KM500	Analysis of Trace Metals by Graphic Furnace Atomic Absorption Spectrophotometry (GFAA)
KM551	Nephelometric Determination of Turbidity using a Turbidity Meter
KM600	Analysis of Metals by Flame Atomic Absorption Spectrometry
KM1631	Determination of Trace Metals in Water by EPA Method 1631 E
KM6020	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma - Mass Spectroscopy

Semi-Volatiles

KO001	Solution and QC Batch Labeling for Sample Preparation in Semi-Volatile Laboratories
KO005	Semi-Volatile Lab Glassware Washing Procedure
KO504-1	1,2 Dibromomethane (EDB) and 1,2-Dibromo-3-Chloropropane (DBCP) by EPA Method 504.1
KO515-1	The Determination of Chlorinated Phenoxy-acid Herbicides in Drinking Water by EPA Method 515.1
KO515-1P	Herbicide Sample Preparation by EPA Method 515.1
KO525-2	METHOD 525.2 Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry
KO605	Benzidines by HPLC
KO680	Determination of PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry (EPA 680)
KO1664	Hexane Extractable Material (HEM) and Silica Gel-Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry
KO1664.SPE	Hexane Extractable Material (HEM) and Silica Gel-Treated N-Hexane Extract-able Material (SGT-HEM) by Solid Phase Extraction (SPE) and Gravimetry
KO3500B	TurboVap Concentration
KO3510	Separatory Funnel Extraction by Method EPA 3510C
KO3520	Continuous Liquid-Liquid Extraction by Method 3520C
KO3535	METHOD EPA 3535 Solid Phase Extraction (SPE)
KO3545	Accelerated Solvent Extraction (3545)
KO3550	Ultrasonic Extraction by EPA 3550B
KO3550B	Herbicide Soil/ Sludge/Waste, or Wipe Extraction Procedure by EPA Method 3550B
KO3580A	Waste Dilution by EPA Method 3580A
KO3620B	Florisil Cleanup by EPA Method 3620B
KP3660A	Sulfur Cleanup by EPA Method 3660A
KO3665A	Sulfuric Acid/Permanganate Cleanup by EPA Method 3665A

KO8011	For 1,2-Dibromomethane (EDB) and 1,2-Dibromo-3-Chloropropane (DBCP) by EPA Method 8011
KO8015FID1	Methanol and Ethanol by Gas Chromatography and Flame Ionization Detection (Method 8015)
KO8015FID2	Miscellaneous Compounds by Gas Chromatography and Flame Ionization Detection (Method 8015)
KO8015FID3	Ethylene and Propylene Glycol by Gas Chromatography and Flame Ionization Detection (Method 8015)
KO8015-NPD1	Nitrogen Containing Compounds by Gas Chromatography and Nitrogen Phosphorous Detection (Method 8015)
KO8015-NPD2	Triethylamine Low Level by Gas Chromatography and Nitrogen Phosphorous Detection (Method 8015)
KO8081A	Analysis of Pesticides by EPA Method 8081A
KO8082	Analysis of PCB by EPA Method 8082
KO8082-oil	Extraction of PCB's in oil
KO8150-DZO	Diazomethane Generation using MNNG and Sodium Hydroxide for Methods 515.1 and 8151A
KO8151	Analysis of Herbicides by EPA Method 8151A
KO8151A-PRE	Herbicide Sample Preparation by EPA Method 8151A
KO8270C	Semi-Volatile Compounds by GC/MS (8270C)
KO8270C.LVI	Semi-Volatile Compounds by Large Volume Injection and GC/MS (8270C)
KO8270-DRO	Diesel Range Organics - Total Petroleum Hydrocarbons by GC-MS (Method 8270)
KO8315.1667	Aldehyde Sample Preparation and Analysis by High Performance Liquid Chromatography (HPLC)
KOBiocide	2(3H)Benzothiazoethione (Biocide) Sample Preparation and Analysis by High Performance Liquid Chromatography (HPLC)
KODZO	Diazomethane Generation using Diazald and Potassium Hydroxide for Methods 515.1 and 8151A
KOTO4PCB	Method for the Determination of Polychlorinated Biphenyls in Ambient Air by EPA TO4
KOWipe	Surface Wipe Extraction for PCB Analysis

Volatile Organics

KO524CAL	Calibration of Hewlett-Packard Gas Chromatograph/Mass Spectrometer with Purge and Trap
KO524MS	Determination of Volatile Organics by GC/MS by 524.2 using HP GC-MS
KO5035H	Closed - System Purge & Trap and Extraction for High-Level Volatile Organics
KO8260HP	The Determination of Volatile Organics by 8260B using Hewlett Packard GC/MS

KO9060B	Analysis of Non-Purgeable Organic Carbon (NPOC)
KODioxane	Determination of 1,4-Dioxane by 8260B using Hewlett Packard GC/MS
KOTO-14	Determination of Toxic Organic Compounds in Ambient using Specially Prepared Canisters with Subsequent analysis by Finnigan ITD GC/MS
KOTO-14F	Sampling Toxic Organic Compounds in Ambient Air using Specially Prepared Canisters
KOTPH-GROHP	Determination of Gasoline Range Organics by GC/MS by 8260B using H-P 5972B GC-MS

Waste

KW005	The ASTM Water-Shake Extraction of a Solid Waste
KW010	The Determination of Heat of Combustion Using Bomb Calorimetry
KW015	The Boiling Point Determination
KW025	The Evaluation of a Solid Waste for the Characteristic of Ignitability
KW030	The Determination of Flash Point using the Pensky-Martens Closed Cup Apparatus
KW035	The Evaluation of a Sample for Corrosivity
KW040	The Determination of Specific Gravity and/or Density
KW045	The Classification and Assignment of a Packing Group to a Readily Combustible Solid of Division 4.1 Using the Burning Rate Test
KW050	The Quantitation of Total Halogens Via Bomb Combustion
KW060	The Evaluation of Solid Waste for the Characteristic of Reactivity (Includes Procedure for Interaction with Water)
KW065	Leaching Procedures: TCLP, SPLP and Method 8315
KW070	The Determination of Total Sulfur
KW080	The Determination of Viscosity Using the Brookfield Viscometer
KW090	Determination of Water Content Using a Karl Fischer Titrimeter
KW100	The Characterization of Materials of Unknown Composition
KW200	The Management of Laboratory Waste: Organic Solvents
KW210	The Management of Laboratory Waste: PCB-Contaminated Materials
KW220	The Management of Laboratory Waste: Client Samples

Water Quality

KQ010	The Calibration of Temperature Measuring Devices Against an NIST - Traceable Thermometer
KQ027	Policies and Practices for Proper Dilution
KQ037	The Operation and Care of the Precision DD-20 Vacuum Pump

KQ040	Recordkeeping Associated with the Preparation of Reagents for Water Quality Analysis
KQ042	The Preparation of Eh Solutions
KQ062	Use, Care, and Calibration of Mechanical Pipettors in the Water Quality and Trace Metals Laboratories
KQ070	The Cleaning of General Laboratory Glassware and Equipment
KQ085	Determination of Sulfuric and Phosphoric Acid Concentration in an Electropolishing Bath Sample
KQ110	The Aqueous Prep of a Solid Sample for a Water Quality Analysis
KQ115	The Quantitation of Acidity in Water by Titration
KQ117	Total Organic Carbon in Soil by the Walkley-Black Method
KQ120	Quantitation of Alkalinity in Water by Titration
KQ130	The Determination of Biochemical Oxygen Demand (BOD), Carbonaceous Biochemical Oxygen Demand (CBOD), and Ultimate CBOD (UCBOD)
KQ132	Client Notification of Drinking Water MCL Excedence
KQ195	The Quantitation of Residual Chlorine by the DPD Method
KQ200	The Quantitation of Total Residual Chlorine by Iodometric Electrode
KQ205	The Quantitation of Chlorine Demand
KQ210	The Quantitation of Chemical Oxygen Demand (COD) by the Spectrophotometric Method
KQ220	The Determination of Color in Water
KQ222	The Quantitation of Sulfite in Water by Iodometric Titration
KQ230	Monitoring and Maintenance of the Laboratory Water Deionization System
KQ250	The Colorimetric Determination of All Forms of Cyanide
KQ300	The Quantitation of Formaldehyde in Water using the Acetylacetone Method
KQ305	The Semi-quantitative Determination of Heavy Metal Impurities in an Aqueous Sample by Visual Comparison
KQ310	The Determination of Hardness by Calculation
KQ311	The Determination of Total Hardness in Water by EDTA Titration
KQ330	The Determination of Water's Saturation (Langelier) Index
KQ340	The Analysis of Methylene Blue-Active Substances (MBAS) in Water
KQ355	The Quantitation of Ammonia-Nitrogen in Water by Flow Injection Analysis
KQ356	The Quantitation of Ammonia-Nitrogen in Water by Discrete Analysis
KQ365	The Quantitation of Nitrate-Nitrogen and/or Nitrite-Nitrogen in Water using the Lachat QuikChem AE(FIA)
KQ375	The Quantitation of Oil and Grease (as Syltherm)
KQ377	The Determination of Visual Oil and Grease (NDNR)
KQ380	The Quantitation of Chloride in Water by Flow Injection Analysis

KQ381	The Quantitation of Chloride in Water by Discrete Analysis
KQ392	The Determination of Total Kjeldahl Nitrogen by Thermal Digestion and Flow Injection Analysis
KQ395	The Quantitation of Total Nitrogen in an Aqueous Sample
KQ396	The Quantitation of Organic Nitrogen in an Aqueous Sample
KQ397	The Quantitation of Total Inorganic Nitrogen in an Aqueous Sample
KQ405	Determination of Threshold Odor in Water
KQ410	The Quantitation of Dissolved Oxygen in Water by Membrane Electrode
KQ425	The Determination of Soil pH and/or Waste pH by the Electrometric Method
KQ430	The Determination of Total Phenolics in Water by Distillation and Flow Injection Quantitation
KQ440	The Colorimetric Determination of Hexavalent Chromium in Water
KQ441	Alkaline Digestion for Hexavalent Chromium in Soil and Solids by EPA 3060A
KQ445	The Quantitation of Permanganate in Water
KQ450	The Colorimetric Determination of Various Forms of Phosphorus
KQ455	The Determination of Phosphorus Adsorption Capacity and Bray P1 Concentration of a Soil
KQ461	The Analysis of Silica and/or Silicate by the Heteropoly Blue Method
KQ462	The Gravimetric Determination of Moisture and Total Solids at 103-105°C
KQ463	The Determination of Dry Weight Solids at 103-105°C
KQ464	The Gravimetric Determination of Fixed and/or Volatile Solids at 550°C
KQ466	The Gravimetric Determination of Total, Fixed and/or Volatile Dissolved Solids at 180°C
KQ468	The Gravimetric Determination of Total, Fixed, and Volatile Suspended Solids
KQ500	The Quantitation of Volatile Organic Acids in Water by Distillation and Titration
KG535	The Quantitation of Total and Reactive Sulfide by Iodometric Titration
KQ575	The Determination of Water-Soluble Substances
KQ585	The Quantitation of Tannin and Lignin in Water
KQ590	Operation of the Lachat QuikChem AE for Ion Analysis of Flow Injection
KQ600E	Determination of Fluoride, Chloride, Nitrite, Nitrate, Bromide, Phosphate and/or Sulfate in Water by Ion Chromatography
KQ610	Operation and Maintenance of the SmartChem® Discrete Analyzer
KQ620	The Operation and Calibration of the Perkin Elmer 1605 FT-IR

Analysis of Water Samples for the
Safe drinking Water Act

The following methods are used for the analysis of drinking water for Safe Drinking Water Act Compliance Monitoring:

Microbiological:
- Total Coliform - Membrane Filter Technique & Presence/Absence test
- Fecal Coliform - EC Broth Technique; Membrane Filter Technique
- Heterotrophic Plate Count - Poured Plate Technique

Inorganic Chemistry:
- Antimony - U.S. EPA Method 200.8
- Arsenic - U.S. EPA Method 200.8
- Barium - U.S. EPA Method 200.8
- Beryllium - U.S. EPA Method 200.8
- Cadmium - U.S. EPA Method 200.8
- Chromium - U.S. EPA Method 200.8
- Copper - U.S. EPA Method 200.8
- Cyanide - U.S. EPA Method 335.2
- Fluoride - Standard Methods EPA 300.0A
- Lead - U.S. EPA Method 200.8
- Mercury - U.S. EPA Method 245.2
- Nickel - U.S. EPA Method 200.8
- Nitrate - U.S. EPA Method 353.2
- Nitrite - U.S. EPA Method 353.2
- Selenium - U.S. EPA Method 200.8
- Sulfate - U.S. EPA Method 300.0
- Sodium - U.S. EPA Method 200.7
- Thallium - U.S. EPA Method 200.8

Organic Chemistry:
- Total Trihalomethanes, Regulated and Unregulated - US EPA Method 524.2
- Volatile Organic Chemicals, Vinyl Chloride - U.S. EPA Method 524.2
- Dibromochloropropane, Ethylene Dibromide - U.S. EPA Method 504.1
- Alachlor, Aldrin, Atrazine, Benzo(a)pyrene, Butachlor, Chlordane, Dieldrin, Di(2-ethylhexyl)adipate, Di(2-ethylhexyl)phthalate, Endrin, Heptachlor, Heptachlor Epoxide, Hexachlorobenzene, Hexachlorocyclopentadiene, Lindane, Methoxychlor, Metolachlor, Metribuzin, Propachlor, Simazine, Toxaphene - U.S. EPA Method 525.2
- Dalapon, Dinoseb, Dicamba, Pentachlorophenol, Picloram, 2,4-D, 2,4,5-TP - U.S. EPA Method 515.2

Drinking water samples have special requirements under the Safe Drinking Water Act regulations. Quality control requirements are both regulatory and method specific. Analysts are reminded of those QC requirements when entering results in LIMS. Special reporting requirements are required for drinking water samples that exceed MCLs. These requirements can be found in specific SOPs but generally require notifying a supervisor in the event of an MCL failure. All samples with a sample type designated “SDWA” require that only the above methods be used for analysis and all other SDWA requirements apply.

APPENDIX H

QC REVIEW FORM

Project No: [redacted]

Requested By: _____ **Date** _____ **Time** _____ **Through:** _____
(Project Manager) (@KAR)

_____ **Phone #** (____) _____-_____
(Company name)

Lead Investigator: **Mike J.** **Bill R.** **Dave A.**

Specific question or focus of this inquiry: _____

_____

_____

_____

Investigative:

Log-in OK?	☐Yes	☐ No	☐ Not applicable
Sample Labeled Correctly?	☐Yes	☐ No	☐ Not applicable
Raw data in order and presentable?	☐Yes	☐ No	☐ Not applicable
Math correct?	☐Yes	☐ No	☐ Not applicable
System clean (Lab Method Blank)?	☐Yes	☐ No	☐ Not applicable
Initial calibration acceptable?	☐Yes	☐ No	☐ Not applicable
Calibration verified?	☐Yes	☐ No	☐ Not applicable
Precision and accuracy verified?	☐Yes	☐ No	☐ Not applicable

Findings: _____

_____

_____

_____

_____

_____

Client was contacted on: Date _____ **Time** _____ **by** _____ **with the following information:**

_____

_____

_____

_____

Inquiry resolved/closed: _____ **Date** _____
(QA Officer)

This form is required under Section 8.1 of the KAR Quality Assurance Manual. File the completed form with the original project paperwork. ***Our goal: Initial response within 8 working hours.***
F:\WPDOCS\QA Manual and Forms\QC Review Form.wpd

APPENDIX I

Initial Demonstration of Capability (IDC)
Training Record Certificate

TEST : _____
METHOD : _____ KAR SOP: _____
ANALYST: _____

Training Requirements:
Performed wherever applicable & supporting documentation attached, if applicable.

_____Analyst has read the Reference Method and understands its contents.
Initials & Date

_____Analyst has read the KAR SOP and understands its contents.
Initials & Date

_____Analyst has prepared and/or analyzed successfully (✓ those that apply):
Initials & Date

- ☐ **Lab Method Blank**
- ☐ **Method Detection Limit (MDL)**
- ☐ **Calibration standard(s)**
- ☐ **Check Standard (Value: _____ Measured: _____ % Rec. _____)**
- ☐ **Performance Evaluation sample (unknown sample)**

Training performed by: _____ Date: _____
Training consisted of:

Comments:

Analyst signature: _____ Date: _____

The analyst has demonstrated proficiency in performing this method.

Manager: (print)_____

Signature: _____Date: _____

Quality Assurance : (print) _____

Signature: _____Date: _____

APPENDIX J

ARCADIS BBL

Attachment 1-3

KAR Organic SOPs

STANDARD OPERATING PROCEDURE

Analysis of PCB by EPA Method 8082

1. References:

1.1 "Test Methods for Evaluating Solid Waste" - SW-846, 3rd Ed., Update III, U.S.EPA, December 1996.
1.2 "Analysis of Pesticides by EPA Method 8081A," KAR Laboratories, Inc., SOP KO8081A.
1.3 "Separatory Funnel Extraction by EPA Method 3510C," KAR Laboratories, Inc., SOP KO3510.
1.4 "Accelerated Solvent Extraction (3545)," KAR Laboratories, Inc., SOP KO3545.
1.5 "METHOD EPA 3535 Solid Phase Extraction (SPE)," KAR Laboratories, Inc., SOP KO3535.
1.6 "Waste Dilution by EPA Method 3580A," KAR Laboratories, Inc., SOP KO3580A.
1.7 "Turbovap Concentration," KAR Laboratories, Inc., SOP KO3500B.
1.8 "Sulfur Cleanup by EPA Method 3660A," KAR Laboratories, Inc., SOP KO3660A.
1.9 "Florisil Cleanup by EPA Method 3620B," KAR Laboratories, Inc., SOP KO3620B.
1.10 "Sulfuric Acid/Permanganate Cleanup by EPA Method 3665A," KAR Laboratories, Inc., SOP KO3665A.
1.11 "Analytical Method for the Analysis of Aroclors Exhibit D," EPA CLP SOM01.01, 5/2005.
1.12 "Ultrasonic Extraction by EPA 3550B," KAR Laboratories, Inc., SOP KO3550.
1.13 "Methods For Organic Chemical Analysis of Municipal and Industrial Wastewater", Method 608: Organochlorine Pesticides and PCBs; Appendix A to Part 136.

2. Scope & Application:

2.1 Method 8082 is used to analyze environmental samples and determine the concentration of polychlorinated biphenyls (PCBs) as Aroclors. Open-tubular capillary columns are employed with an electron capture detector (ECD). When compared to packed columns, these fused-silica open-tubular columns offer improved resolution, better selectivity, increased sensitivity and faster analysis. The nine PCB Aroclors listed in Section 2.2 are analyzed using a dual-column analysis system.

2.2

Analyte	CAS Registry No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

2.3 PCB Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies. Such weathered multi-component mixtures may have significantly different peak patterns than those of Aroclor standards.

2.4 Quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment.

2.5 GC/MS Method 8270C is recommended as a confirmation technique when sensitivity permits.

2.6 This method utilizes a dual-column system configuration of two analytical columns joined to a single injection port.

2.7 The analyst must select columns, detectors and calibration procedures most appropriate for the analytes of interest. Matrix-specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix (e.g., hexane solutions from sample extractions, diluted oil samples, etc.). Sample chromatograms for the predominant seven PCB Aroclors are provided as attachments to this SOP. GC operating conditions are specified in Section 10.4.

2.8 The reporting limit for each Aroclor in water is 0.10 µg/L. The reporting limit in soil is 330 µg/kg (on a dry weight basis) for each Aroclor. These reporting limits are highly matrix-dependent and may be elevated when a complex sample type is encountered.

2.9 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GC) and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method by an initial demonstration of proficiency.

3. Summary of Method:

3.1 A measured volume or weight of sample (approximately 1 L for most waters, 2 g to 30 g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

3.2 Aqueous samples are extracted at neutral pH with methylene chloride using a separatory funnel (refer to KAR SOP KO3510).

3.3 Solid samples are extracted with methylene chloride using pressurized fluid extraction ("accelerated solvent extraction"), according to KAR SOP KO3545.

3.4 Extracts for PCB analysis may be subjected to various cleanup procedures including mercury treatment (Section 10.3.1), florisil cleanup (Section 10.3.1) and sulfuric acid/potassium permanganate cleanup (Section 10.3.1). These cleanup techniques may remove or destroy certain single component organochlorine or organophosphorus pesticides. Therefore, Method 8082 is not applicable to the analysis of those compounds. Instead, use Method 8081 (KAR SOP KO8081A).

3.5 Following cleanup, the extract is analyzed by injecting a 2 µL aliquot into a gas chromatograph with dual wide-bore fused silica capillary columns and electron capture detectors (GC/ECD).

3.6 The chromatographic data may be used to determine concentrations of each of the nine Aroclors listed in Section 2.2.

4. Interferences:

4.1 Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into three broad categories:

- Contaminated solvents, reagents, or sample processing hardware.

- Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.
- 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. 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 through the analysis of laboratory method blanks and/or reagent blanks. Generally, any phthalates present in a raw sample extract may be removed through the use of florisil cleanup (Section 10.3.1).

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 13° C for several hours, or rinse with methanol and drain. Store dry glassware (covered) 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, do not dry glassware used 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. Sulfur is generally removed by using mercury cleanup (Section 10.3.1).

5. Apparatus & Equipment:

5.1 Tremetrics 9001 GC with dual Electron Capture Detectors, Leap Technologies CTE-A200SE Autosampler and ChromPerfect data system with Report Write Plus PCB software.
GC columns: J&W Scientific DB-5 (30 m x 0.53 mm ID, 1.5µm film thickness) and J&W Scientific DB-608 (30 m x 0.53 mm ID, 0.83 µm film thickness).
Guard Column: 10 m section of uncoated deactivated 0.53 mm ID column (Agilent, Cat. No. 160-2535-10).
Column rinsing kit: Bonded-phase column rinse kit (J&W Scientific, Catalog No. 430-3000) or equivalent.
Volumetric flasks, Class A, 10 mL and 25 mL, for preparation of standards.
Syringes, Hamilton, 700 series and gas-tight - 10 µL, 25 µL, 100 µL, 250 µL, 500 µL, 1.25 mL, and 2 mL.
Borosilicate glass vials, 20 mL.
Vials, amber, screw cap, 40 mL.
Vials, glass with polytetrafluoroethylene(PTFE)-lined screw caps or crimp tops, 1 and 20 mL.
Syringe, 10 cc disposable.
Aluminum weighing boat.
Vortex mixer.
Turbovap concentrating tubes and rack.
Pasteur Pipets, 6" disposable.
Florisil Cartidges, Waters Sep-Pak Plus, Grade "A" (Waters Part No. WAT020525).
Disposable pipet, graduated, 5 mL.
Pipet bulbs.
Teflon squeeze bottle, 500 mL.
Graduated cylinder, glass, 500 mL.
Griffin beaker, glass, 1000 mL.

Erlenmeyer Flask, glass, 1000 mL.
Glass jug, amber, 4 L.

6. Sample Handling:

6.1 PCBs in Water: Store in a glass container with Teflon-lined cap at 4°C. Holding times are 7 days until extraction and 40 days for analysis of extract stored in sample freezer.

6.2 PCBs in Soil: Store in a glass container with Teflon-lined cap at 4°C. Holding times are 14 days until extraction and 40 days for analysis of extract stored in sample freezer.

7. Reagents:

7.1 Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 NOTE: Store the standard solutions (stock, composite, calibration, internal and surrogate standards) at 4°C in polytetrafluoroethylene (PTFE)-sealed containers in the dark. All stock standard solutions must be replaced within one year; replace standards sooner if routine QC indicates a problem. All other standard solutions must be replaced within six months, or earlier if routine QC indicates a problem.

7.3 Sample extracts prepared by Methods 3510, 3520, 3545 or 3550 need to undergo a solvent exchange step prior to analysis. The following solvents are necessary for solvent exchange and/or dilution of sample extracts. All solvents should be pesticide quality or equivalent and should be determined to be phthalate-free through the analysis of laboratory method blanks and/or reagent blanks.

n-Hexane, C_6H_{14}.
Isooctane, $(CH_3)_3CCH_2CH(CH_3)_2$.

7.4 The following solvents may be required for the preparation of standards. All solvent lots must be pesticide quality or equivalent and should be determined to be phthalate-free through the analysis of laboratory method blanks and/or reagent blanks.

Acetone, $(CH_3)_2CO$.
Toluene, $C_6H_5CH_3$.

7.5 The following reagents and materials may be required for sample extraction or cleanup:

Sulfuric acid, H_2SO_4, concentrated, ACS grade.
Potassium permanganate, $KMnO_4$, crystals, ACS grade.
Mercury, triple-distilled.
Hexane, C_6H_{14} - Pesticide quality or equivalent.

Sulfuric acid/water, H_2SO_4/H_2O (1:1, v/v):
Slowly add, from a 500 mL glass graduated cylinder, 250 mL of concentrated H_2SO_4 to 250mL organic-free reagent water in a 1000 mL glass beaker. Caution! The reaction will generate a significant amount of heat. Store at room temperature for up to one year.

Potassium permanganate, $KMnO_4$ (5 percent aqueous solution, w/v):
Weigh 50 g of $KMnO_4$ into an aluminum weighing boat and add to 1000 mL of organic-free reagent grade water in a 1000 mL Erlenmeyer flask. Using a magnetic stir bar and stir plate, mix until the $KMnO_4$ is dissolved. Store in a 4 L amber glass jug. Store at room temperature

for up to one year.

Florisil Cartridges, Waters Sep-Pak Plus, Grade "A" (Waters Part No. WAT020525).

8. Preparation of Standards:

8.1 Stock standard solutions

Aroclor 1016 (Supelco, Cat. No. 4-8097), 1000 µg/mL in isooctane
Aroclor 1221 (Supelco, Cat .No. 4-8098), 1000 µg/mL in isooctane
Aroclor 1232 (Supelco, Cat. No. 4-4805), 1000 µg/mL in isooctane
Aroclor 1242 (Supelco, Cat .No. 4-4806), 1000 µg/mL in isooctane
Aroclor 1248 (Supelco, Cat .No. 4-4807), 1000 µg/mL in isooctane
Aroclor 1254 (Supelco, Cat .No. 4-4808), 1000 µg/mL in isooctane
Aroclor 1260 (Supelco, Cat. No. 4-4809), 1000 µg/mL in isooctane
Aroclor 1262 (Supelco, Cat. No. 4-4810), 1000 µg/mL in isooctane
Aroclor 1268 (Chemservice, Cat. No. F295S), 1000 µg/mL in methanol

8.2 Surrogate standard solution: Pesticide Surrogate Spike Mix (Supelco, Cat.No. 4-8460)
200 µg/mL each component in acetone:
Decachlorobiphenyl
Tetrachloro-m-xylene

8.3 Intermediate stock solutions

8.3.1 Aroclor 1016/1260 Mix (50 µg/mL each in hexane). Dilute 0.500 mL of each 1000 µg/mL stock standard to 10 mL with hexane using 1000 µL Hamilton syringe and 10 mL class A volumetric flask. Store in 20mL glass vial with PTFE-lined screw cap and place in the standards freezer.

8.3.2 Aroclor 1221, 1232, 1242, 1248, 1254, 1262 and 1268 (individual) stock solutions (50 µg/mL in hexane). Dilute 0.500 mL of 1000 µg/mL stock standard to 10mL with hexane using a 1000 µL Hamilton syringe and 10 mL class A volumetric flask. Store all individual solutions in 20 mL glass vials with PTFE-lined screw caps and place in the standards freezer.

8.3.3 Surrogate standard (20 µg/mL each in hexane). Dilute 1.00 mL of 200 µg/mL stock surrogate mix to 10 mL with hexane using a 1.25 mL Hamilton syringe and 10 mL class A volumetric flask. Store in 20 mL glass vial with PTFE-lined screw cap and place in the standards freezer.

8.4 Surrogate spiking solutions

8.4.1 To prepare aqueous surrogate spiking solution (2 µg/mL each in acetone): Dilute 0.250 mL of 200 µg/mL surrogate spike mix to 25 mL with acetone using a 500 µL Hamilton syringe and 25 mL class A volumetric flask. Using a 250 µL syringe, add 100µL of aqueous surrogate spiking solution to 1 L of sample to create a 0.20 µg/L spike. Store the aqueous surrogate spiking solution in a 40 mL amber glass vial with a PTFE-lined screw cap and place in the standards freezer.

8.4.2 To prepare soil surrogate spiking solution (20 µg/mL in acetone): Dilute 1.00 mL of 200 µg/mL surrogate spike mix to 10 mL with acetone using a 1.25 mL Hamilton syringe and 10 mL class A volumetric flask. Using a 250 µL syringe, add 100 µL of soil surrogate spiking solution to 10 g of sample to create a 200 µg/kg spike. Store the soil surrogate spiking solution in a 20 mL glass vial with a PTFE-lined screw cap and place in the standards freezer.

8.5 PCB (Aroclor) working solutions

8.5.1 Aroclor 1016/1260 mix calibration standards (0.10, 0.30, 0.50, 1.00, 1.5, and 2.00 µg/mL). Add 0.10, 0.30, 0.50, 1.00, 1.5, and 2.00 mL respectively of the 10 µg/mL Aroclor 1016/1260 mixture to each of six 10 mL class A volumetric flasks. The surrogates are then added to each of the Aroclor 1016/1260 calibration standards at 0.05, 0.10, 0.20, 0.30, 0.35, and 0.40 µg/mL each, respectively. This is accomplished by adding 25, 50, 100, 150, 175, and 200 µL, respectively, of the 20 µg/mL intermediate surrogate solution to each of the six 10 mL volumetric flasks. Dilute each solution to 10 mL with hexance.

NOTE: Each of the six Aroclor 1016/1260 working solutions are used for initial calibration. The Aroclor 1016/1260 0.50 µg/mL working standard is also used for continuing calibration. See Section 10.8.3 for the acceptance criterion related to continuing calibration verification.

8.5.2 Aroclors 1221, 1232, 1242, 1248, 1254, 1262 and 1268 (individual) working solutions (0.50 µg/mL each). Add 0.50 mL of the 10 µg/mL intermediate stock and dilute to 10 mL with hexane using a 1000 µL Hamilton syringe and a 10 mL class A volumetric flask.

NOTE: In addition to the six Aroclor 1016/1260 calibration standards, each of the (individual Aroclor 1221-1254) 0.50 µg/mL working solutions is analyzed at the time of initial calibration. Because only a single-point calibration is performed for each of these other Aroclors, no RSD performance criterion is applied. Aroclors 1262 and 1268 are only analyzed when requested by the client as additional compounds.

8.6 Quality control check (secondary source) standards and spiking solutions

8.6.1 Aroclor 1221 (Restek Cat. No. 32007), 1000 µg/mL in hexane
Aroclor 1232 (Restek Cat. No. 32008), 1000 µg/mL in hexane
Aroclor 1242 (Restek Cat. No. 32009), 1000 µg/mL in hexane
Aroclor 1248 (Restek Cat. No. 32010), 1000 µg/mL in hexane
Aroclor 1254 (Restek Cat. No. 32011), 1000 µg/mL in hexane
Aroclor 1016/1260 (Restek Cat. No. 32039), 1000 µg/mL in hexane

8.6.2 All Aroclor quality control check standards are prepared at 0.50 µg/mL in hexane. The Aroclor 1016/1260 QC check standard is analyzed with the continuing calibration standard every 12-hour shift. This additional quality control measure serves to confirm the accuracy of the Aroclor calibration standards. The results for both standards must satisfy the acceptance criterion for continuing calibration verification (see Sections 10.8.3 and 10.8.4). Consult Sections 8.3 and 8.5 for information regarding the preparation of 0.50 µg/mL standards.

8.7 Quality control check (secondary source) surrogate standards and spiking solutions

8.7.1 Pesticide surrogate mix (Restek Cat. No. 32000), 200 µg/mL each in acetone:
Decachlorobiphenyl
2,4,5,6-tetrachloro-m-xylene

8.7.2 The QC surrogates are added to the Aroclor 1016/1260 0.50 µg/mL QC check standard and each individual Aroclor 0.50 µg/mL QC check standard at 0.20 µg/mL each. Consult Sections 8.3 and 8.5 for information regarding the preparation of 0.50 µg/mL standards.

8.8 PCB (Aroclor) spiking solution (prepared from QC check standards)

8.8.1 Aqueous PCB spiking solutions (5 µg/mL each Aroclor 1016/1260 and Aroclor 1242). Dilute 0.125 mL of 1000 µg/mL Aroclor stock solution to 25mL with hexane using a 250 µL syringe

and 25 mL class A volumetric flask. Transfer the aqueous PCB spiking solution to a 40 mL amber vial and store in the standards freezer.

8.8.2 Using a 200 µL syringe, add 100 µL of 5 µg/mL aqueous PCB spiking solution to 1 L of sample to create a 0.50 µg/L spike.

8.8.3 Soil PCB spiking solutions (25 µg/mL each Aroclor 1016/1260 and Aroclor 1242). Dilute 0.625 mL of 1000 µg/mL Aroclor stock solution to 25mL with hexane using a 1000 µL syringe and 25 mL class A volumetric flask. Transfer the soil PCB spiking solution to a 40 mL amber vial and store in the standards freezer.

8.8.4 Using a 1.0 mL syringe, add 0.500 mL of 25 µg/mL soil PCB spiking solution to 10 g of sample to create a 1250 µg/kg spike.

8.9 Calibration standards for PCB Aroclors

8.9.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five PCB Aroclors. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at six concentrations is sufficient to demonstrate the linearity of the detector response without the need to perform initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentration of Aroclor 1016 or Aroclor 1260 present in a sample.

8.9.2 According to the instructions in Section 8.5.1, prepare six calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260 by dilution of the stock standard with hexane. These concentrations correspond to the expected range of concentrations generally found in actual samples and bracket the linear range of the detector.

8.9.3 Single standards of each of the other six Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards described above have been used to demonstrate the linearity of the detector, these single standards of the remaining seven Aroclors are also used to determine the calibration factor for each Aroclor. Prepare a (0.50 µg/mL) standard for each of the other Aroclors following the instructions in Section 8.5.2. This concentration corresponds to the approximate midpoint of the linear range of the detector.

9. Safety:

9.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. A lab coat, gloves and safety glasses or a face shield should be worn while handling extracts and standards. Standard preparation, addition of the internal standard solution, and sample extract dilution must be performed in a hood.

9.2 Material Safety Data Sheets (MSDS) are available to all laboratory employees. These sheets specify the type of hazard that each chemical poses and the procedures that are used to handle these materials safely.

9.3 Due to the hazardous nature of PCBs, extreme measures must be taken to ensure proper disposal of all PCB contaminated samples and/or reagents. ANY PCB RESULT $\geq$ 50 PPM MUST IMMEDIATELY BE REPORTED TO THE DEPARTMENT SUPERVISOR OR DESIGNEE. The supervisor is then responsible for collecting all affected samples/extracts and forwarding them to the waste manager for

proper disposal. SAMPLES CONTAINING PCBS ≥ 50 PPM MUST NEVER BE POURED DOWN THE DRAIN.

10. Procedure:

10.1 Sample extraction

10.1.1 Refer to the following KAR SOPs for the appropriate matrix and extraction technique:

Aqueous	KO3535: Solid Phase Extraction
Aqueous, TCLP Extracts	KO3510: Separatory Funnel
Soils/Sediments	KO3545: Accelerated Solvent Extraction
Wastes	KO3580A: Waste dilution

10.2 Extract concentration

10.2.1 Refer to KAR SOP KO3500B.

10.3 Extract cleanup

10.3.1 Refer to KAR SOP KO3660A for sulfur cleanup, KP3620B for florisil cleanup, and KO3665A for sulfuric acid/permanganate cleanup.

10.4 GC operating conditions:

Column 1: DB-5 (30 m x 0.53 mm ID, 1.5 µm film thickness)
Column 2: DB-608 (30 m x 0.53 mm ID, 0.83 µm film thickness)
Guard column: (J&W) 10 m x 0.53 mm ID, deactivated fused silica
Carrier gas (Hydrogen) flow rate: 6 mL/min
Makeup gas (Nitrogen) flow rate: 60 mL/min total flow
Temperature program:
a) 0.5 min hold at 150° C
b) 150° C to 190° C, at 12° C/min
c) 2.0 min hold at 190° C
d) 190° C to 275° C, at 4° C/min
e) 10.0 min hold at 275° C
f) 37.08 min total run time
Injector temperature: 240° C
Detector temperature: 350° C
Injection volume: 2 µL
Solvent: Hexane
Type of injector: Flash vaporization
Detector type: Dual ECD
Range: 10 volts
Type of splitter: Restek Universal Y-shaped Press-Tight connector

10.5 Calibration

10.5.1 As noted above, a standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other seven PCB Aroclors. Therefore, this standard will be used to establish the linearity of the detector and to demonstrate that a sample does <u>not</u> contain peaks that represent any one of the Aroclors. This standard will also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present

in a sample. A six-point initial calibration will be performed using the mixture of Aroclors 1016 and 1260.

Standards of the other seven Aroclors are necessary for pattern recognition. These standards will also be used to determine a single-point calibration factor for each Aroclor. The standards for these seven Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the six 1016/1260 standards.

In situations where only a few Aroclors are of interest for a specific project, a six-point initial calibration of each of the Aroclors of interest (e.g., six standards of Aroclor 1232 if this Aroclor is of concern) may be employed in place of the 1016/1260 mixture described or the pattern recognition standards described above.

10.5.2 Initial calibration of the system: Inject 2 µL of the 0.50 µg/mL individual Aroclor working solutions in addition to the 0.10, 0.30, 0.50, 1.00, 1.5, and 2.00 µg/mL working solutions of the Aroclor 1016/1260 mixture.

10.5.3 Using the PCB Calculator program, calibrate each Aroclor by "tagging" as many as seven of the most prominent peaks for that particular Aroclor and calculating a calibration factor based on one-point external standard calibration.

A quadratic curve is generated, using the Chrom Perfect data system, which plots the amount versus area for each of the levels and analytes using the external standard method according to criteria in SW-846, method 8000, Section 7. When determining Aroclors by the external standard technique, the calibration factor (CF) for each analyte in each of the initial calibration standards will be calculated using the Chrom Perfect software according to the following equation:

$$CF = \frac{\text{Peak area (or height) in the standard}}{\text{Total mass of the standard injected (in nanograms)}}$$

Calibration factors generated during the initial multipoint calibration are used to establish the linear dynamic range of the analytical system. The GC data system calculates the average calibration factor, the associated standard deviation, the relative standard deviation (RSD), and the correlation coefficient for each Aroclor in the initial calibration mix. The six point quadratic curve with 1/x weighting must have an average correlation coefficient greater than 0.99 for the calibration to be acceptable.

Aroclor 1016 and 1260 concentrations are calculated using the calibration curve generated from the most recent initial calibration. Concentrations of the other Aroclors are calculated using the calibration factor established for that Aroclor at the time of initial calibration. At any time, the analyst may choose to run an appropriate (individual Aroclor) standard to confirm retention times of the Aroclor peaks on both GC analytical columns. However, this does not constitute an attempt at continuing calibration verification; therefore, no RSD criterion need be considered.

10.6 Quantitation of PCBs as Aroclors

10.6.1 The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

10.6.2 Use the individual Aroclor standards (not the 1016/1260 mixtures) to determine the pattern of

peaks for Aroclors 1221, 1232, 1242, 1248, 1254, 1262 and 1268. The patterns for Aroclors 1016 and 1260 will be evident in the mixed calibration standards.

10.6.3 Once the Aroclor pattern has been identified, compare the responses of three to seven major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the peaks selected should include at least one peak that is unique to that Aroclor. A concentration is determined for each of the characteristic peaks using the appropriate individual calibration factor and the equation in Section 10.6.9.1 (water samples) or 10.6.9.2 (soil samples). The resultant (three to seven) peak concentrations are averaged to determine the concentration of the associated Aroclor. Seven peaks are used for the Aroclor 1016/1260 mixture, none of which are common to both of these Aroclors.

10.6.4 Late-eluting Aroclor peaks are generally the most stable in the environment. Table 1 lists thirteen specific PCB congeners found in Aroclor mixtures.

10.6.5 Record the peak area (or height) for each characteristic Aroclor peak to be used for quantitation.

NOTE: Peak area measurements are typically employed for PCB calculations. However, at the analyst's discretion, peak height may be substituted when overlapping peaks make the integration of peak areas difficult. In any case, the use of either peak area or height must remain consistent for an entire batch of standards and samples.

10.6.6 During sample analysis, any extract that produces individual PCB peak areas greater than those in the highest Aroclor 1016/1260 standard must be diluted and reanalyzed until a response that falls within the linear dynamic range (0.10 µg/mL to 2.00 µg/mL) is obtained.

10.6.7 Six sets of calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the seven peaks chosen for this mixture. The single standard for each of the other Aroclors will generate at least three (and as many as seven) calibration factors, one for each selected peak. An Aroclor calibration factor is calculated by averaging all associated peak calibration factors.

10.6.8 The six Aroclor 1016/1260 calibration factors generated during the initial multipoint calibration are used to establish the linear dynamic range of the analytical system. The GC data system calculates the average calibration factor, the associated standard deviation, the relative standard deviation (RSD), and the correlation coefficient for each Aroclor (1016 and 1260) in the initial calibration mix. If the criterion in Section 10.5.3 are satisfied for the initial calibration, then sample analysis may proceed for the remainder of the 12 hour work shift.

10.6.9 Calculation of sample results

The concentration of each analyte in the sample is determined by comparing the detector response (peak area or height) to the response for that analyte in the initial or continuing calibration. The concentration of an analyte may be calculated as follows, depending on the sample matrix:

10.6.9.1 **Aqueous samples**

$$\text{Concentration } (\mu g/L) = \frac{(A_s)\,(V_t)\,(D)}{(\overline{CF})\,(V_i)\,(V_s)}$$

where:

A_s = Area (or height) of the peak for the analyte in the sample.
V_t = Total volume of the concentrated extract (µL).
D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
$\overline{CF}$ = Mean calibration factor from the initial calibration (area per ng).
V_i = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.
V_s = Volume of the aqueous sample extracted in mL. If units of liters are used for this term, multiply the results by 1000.

Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.

10.6.9.2 **Nonaqueous samples**

$$\text{Concentration } (\mu g/kg) = \frac{(A_s)(V_t)(D)}{(\overline{CF})(V_i)(V_s)}$$

where A_s, V_t, D, $\overline{CF}$, and V_i are as described in 10.6.9.1, and

W_s = Weight of sample extracted (g). Although this term is generally expressed as dry weight, either the wet weight or dry weight may be used, depending upon the specific application of the data. When dry weight reporting is desired, W_s is equivalent to the wet weight of extracted sample multiplied by the percent solids (expressed as a decimal): $W_s = W_{wet} \times$ (% solids/100). Refer to KAR SOP KG462 for details regarding percent solids determination.

Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.

10.6.10 Quantitation notes:

Use only peaks appearing in both the standard and the sample for calculations.

Round the calculated concentration to two significant figures except when the results are less than ten times the reporting limit. If this occurs, report only one significant figure.

The reporting limit in the absence of interferences will be 0.1 µg/L for water samples and 330 µg/kg (on a dry weight basis) for soils. Identify any Aroclor detected on the analytical report.

10.6.11 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. In such instances, if results in terms of Aroclors are absolutely required, then the quantitation as Aroclors may be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs on the basis of retention times should be subtracted from the total area. When quantitation is performed in this manner, the problems should be fully described for the data user and the specific procedures employed by the analyst should be thoroughly documented.

10.6.12 GC/MS confirmation may be used in conjunction with dual-column analysis if the concentration is sufficient for detection by GC/MS.

10.6.13 Full-scan quadrupole GC/MS will normally require a higher concentration of the analyte of interest than full-scan ion trap or selected ion monitoring techniques. The concentrations will be instrument-dependent, but values for full-scan quadrupole GC/MS may be as high as 10 ng/μL in the final extract, while ion trap or SIM may only require a concentration of 1 ng/μL.

10.6.14 The GC/MS must be calibrated for the specific target analytes. When using SIM techniques, the ions and retention times should be characteristic of the Aroclors to be confirmed.

10.6.15 GC/MS confirmation should be accomplished by analyzing the same extract used for GC/ECD analysis and the extract of the associated blank.

10.6.16 The base/neutral/acid extract and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere. However, if PCBs are *not* detected in the base/neutral/acid extract, then GC/MS analysis of the pesticide extract should be performed.

10.7 Retention time windows

10.7.1 Absolute retention times will be used for the identification of Aroclor peaks. A retention time window study must be performed whenever new column(s) are installed or whenever significant changes and/or major maintenance are performed on the gas chromatographs. This study consists of 3 standard injections over a 72 hour period and calculation the retention time window of all surrogates, and major constituents of multi-component analytes (PCBs) as ± 3 times the standard deviation of the mean absolute retention time established over the 72 hour period. These windows must be calculated, documented , and readily accessible for easy reference.

10.7.2 Monitor retention time stability by comparing the absolute retention times of analyte peaks in both initial and continuing calibration standards. Acceptance criteria are expressed as the maximum acceptable retention time shift, in minutes. A warning limit of ±0.10 minutes retention time shift has been established for PCB analysis at KAR Laboratories. If the retention times vary by more than the windows established according to the procedure in sec. 10.7.1, then a new set of calibration standards must be analyzed and the system must be recalibrated.

10.7.3 For Aroclors other than 1016 or 1260, the analyst may wish to analyze the appropriate (individual Aroclor) standard following an apparent positive sample result. If so, the chromatographic pattern and major peak retention time data from both analytical GC columns may be used to aid Aroclor identification and confirmation.

10.8 Gas chromatographic analysis of sample extracts

10.8.1 Identical GC operating conditions must be employed for both instrument calibration and the analysis of samples.

10.8.2 Verify calibration each 12-hour shift by injecting the (0.50 μg/mL Aroclor 1016/1260 mix) continuing calibration standard prior to conducting any sample analyses. The (0.50 μg/mL Aroclor 1016/1260 mix) QC check standard is analyzed with the continuing calibration standard every 12-hour shift.

A continuing calibration standard must also be injected at intervals of not less than once every twenty samples (after every 10 samples is recommended to minimize the number of samples

requiring reinjection when QC limits are exceeded) and at the end of the analysis sequence. For Aroclor analyses, the continuing calibration verification standard should be a mixture of Aroclor 1016 and Aroclor 1260. The calibration verification process does not *require* analysis of the other Aroclor standards used for pattern recognition, but the analyst may wish to include a standard for one of these Aroclors after the 1016/1260 mixture used for calibration verification throughout the analytical sequence.

10.8.3 The calculated amount for each analyte in the continuing calibration from the initial calibration curve (CA_c) must not exceed a difference of more than ± 15 percent when compared to the true value (TV).

$$\%\,Difference = \frac{|(TV - CA_c)|}{TV} \times 100$$

10.8.4 Similarly, the calculated amount for each analyte calculated from the QC check standard (CA_{QC}) must not exceed a difference of more than ± 15 percent when compared to the true value (TV).

$$\%\,Difference = \frac{|(TV - CA_{QC})|}{TV} \times 100$$

10.8.5 If the 15 percent criterion is exceeded for any (continuing calibration standard or QC check standard) calibration factor, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.8.6 If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed.

10.8.7 Inject a 2 µL aliquot of the concentrated sample extract. Record the volume injected to the nearest 0.05 µL and the resulting peak size in area (or peak height) units.

10.8.8 Qualitative identifications of target analytes are made by examination of the sample chromatograms, as described in Section 10.9.

10.8.9 Quantitative results are determined for each identified Aroclor using the procedures described in Section 10.6.9. If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyze. The use of peak height rather than peak area in quantitation is recommended when overlapping peaks cause errors in area integration.

10.8.10 Each sample analysis must be bracketed with an acceptable initial calibration, QC check standard and continuing calibration standard(s) (each 12-hour shift) or calibration standards interspersed within the samples. When a continuing calibration or QC check standard fails to meet the QC criteria, all samples that were injected after the most recent standard that met the QC criteria must be reinjected.

10.8.11 Multi-level standards of each Aroclor are not required if the calculated concentration of any Aroclor in the sample extract falls within the 0.10 µg/mL to 2.0 µg/mL linear dynamic range established through initial calibration with the Aroclor 1016/1260 mix.

10.8.12 Sample injections may continue for as long as the continuing calibration standards, QC check standards and standards interspersed with the samples meet instrument QC requirements. At a minimum, standards are analyzed after every 20 samples and/or at the end of an analytical batch. The sequence ends when the entire batch of samples has been injected or when qualitative or quantitative QC criteria are no longer satisfied.

10.8.13 Use the calibration standards analyzed during the sequence to evaluate retention time stability. If the retention time of any of the standards falls outside of the calculated window, the system is out of control. Refer to section 10.10 for potential corrective actions.

10.8.14 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. The analyst may choose to rerun the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to Section 10.3 for the procedures to be followed for sample extract cleanup.

10.9 Qualitative identification

10.9.1 The identification of PCB Aroclors using an electron capture detector is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes.

10.9.2 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: (1) using a second GC column of dissimilar stationary phase (as in the dual-column analysis), (2) based on a clearly identifiable Aroclor pattern or (3) using another technique such as GC/MS .

10.9.3 When simultaneous analyses are performed from a single injection (the dual-column GC configuration), it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are analyzed on both columns, the results for both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed. When reporting results, the higher value of the two columns will be reported as long as they agree within 40% of each other. If not, the chromatograms should be carefully assessed for possible integration errors, calibration errors, or overlapping peak areas. If no errors are found, the lower result should be reported along with advising the data user of the disparity between results on the two columns.

10.10 Chromatographic System Maintenance as Corrective Action

10.10.1 When system performance does not meet the established QC requirements, corrective action is required and may include one or more of the following actions.

10.10.2 Liner and Septa replacement: When retention time shift of 0.15 minutes or more is observed, or chromatography has otherwise deteriorated, replace septa, install clean liner, and remove

approximately 6 inches of guard column. Bake out the system for a few hours at 275°C. It may take 2 to 3 hours for the carrier gas flow to regulate itself. The system is now ready to be recalibrated.

10.10.3 Saturation Current Adjustment: When the baseline of either ECD detector becomes noisy, either the analytical GC columns need to be baked out at 275°C or the saturation current on the ECDs requires readjustment. The saturation current should be set at 35. This current setting is usually very stable, but needs to be readjusted every few years. Consult the Tracor 9001 operating manual for details.

10.10.4 Check of Splitter Connections: For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter. Break off the first few inches of the injection port side of the guard column. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

10.10.5 Inspection of Metal Injector Body: Turn off the oven and remove the analytical columns when the oven has cooled. Remove the glass injection port insert (instruments with on-column injection). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material. Place a beaker beneath the injector port inside the oven. Using a wash bottle, rinse the interior of the injector port with acetone and then rinse it with toluene, collecting the rinsate in the beaker. Consult the manufacturer's instructions regarding deactivating the injector port body. Glass injection port liners may require deactivation with a silanizing solution containing dimethyldichlorosilane.

10.10.6 Column Rinsing: The column should be rinsed with several column volumes of an appropriate solvent. Generally, a series of both polar and nonpolar solvents is recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone. Methylene chloride is a good final rinse and in some cases maybe the only solvent required. The column should then be filled with methylene chloride and allowed to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen.

11. QA/QC:

11.1 KAR Laboratories has established a formal quality assurance program and maintains records to document the quality of all PCB data generated.

11.2 Quality control procedures necessary to evaluate the GC system operation include evaluation of retention time windows (Section 10.7), continuing calibration verification (Section 10.8.3) and chromatographic analysis of samples (Section 10.10).

11.3 Initial Demonstration of Proficiency: KAR has demonstrated initial proficiency (with each sample preparation and determinative method combination) by generating data of acceptable accuracy and precision for target analytes in a clean matrix. Examples of such data are presented in Sections 12.1 through 12.3. The laboratory must also repeat the following operations whenever new analysts are trained or significant changes in instrumentation are made.

11.4 In the absence of recommended acceptance criteria for the initial demonstration of proficiency, as is the case for method 8082, KAR has adopted the range of 70 to130 percent recovery as guidance in

evaluating the results. Given that the initial demonstration is performed in a clean matrix, the average recoveries of analyte from the four to seven replicates should generally fall with this range.

11.5 KAR is generally successful in satisfying the recovery criterion even at analyte concentrations near the reporting limit for the method. Therefore, whenever possible, the initial demonstration of proficiency is performed concurrently with the method detection limit study for a particular analyte.

12. Method Performance:

12.1 PCB Aroclor 1242 Sample Matrix: Aqueous

Replicate	True Value (µg/L)	Measured Value (µg/L)	Grubb's "T" Test (outlier if > 1.938)
1	.0500	.0396	.4499329
2	.0500	.0365	.0562416
3	.0500	.0457	1.224615
4	.0500	.0278	1.048634
5	.0500	.0261	1.264529
6	.0500	.0317	.5533449
7	.0500	.0450	1.135717

Mean:	.0361	% Recovery :	72.11
Std. Dev.:	.0079	Rel. % Diff:	21.84
Signal/Noise:	4.58	MDL:	.024749

12.2 PBC Aroclor 1242 Sample Matrix: Soil

Replicate	True Value (µg/kg)	Measured Value (µg/kg)	Grubb's "T" Test (outlier if > 1.938)
1	200.0	172.8	0.03
2	200.0	181.8	0.56
3	200.0	160.1	0.87
4	200.0	158.6	0.97
5	200.0	191.8	1.23
6	200.0	190.7	1.15
7	200.0	157.2	1.07

Mean:	173.2857	% Recovery:	86.64
Std. Dev.:	15.0944	Rel. %Diff:	8.71
Signal/Noise:	11.48	MDL:	47.441753

12.3 PCB Aroclor 1260 Sample Matrix: Soil

Replicate	True Value (µg/kg)	Measured Value (µg/kg)	Grubb's "T" Test (outlier if > 1.938)
1	200.0	183.8	0.98
2	200.0	169.5	0.96
3	200.0	189.6	1.77
4	200.0	172.9	0.50
5	200.0	174.1	0.34
6	200.0	170.8	0.78
7	200.0	175.3	0.17

Mean:	176.5714	% Recovery:	88.29
Std. Dev.	7.3769	Rel. % Diff.:	4.18
Signal/Noise:	23.94	MDL:	23.185681

12.4 Sample Quality Control for Preparation and Analysis: KAR Laboratories has established procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). These procedures include the analysis of QC samples including a method blank, a matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to all samples, QCs, and blanks.

12.5 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. Generally, KAR uses a matrix spike and matrix spike duplicate pair. However, a laboratory fortified blank and laboratory fortified blank duplicate may be substituted if insufficient sample is available for MS/MSD.

12.6 The following table lists the matrix spike control limits for aqueous and soil matrices:

Compound	% Recovery QC Limits	RPD
AR1016	29-135	0-25
AR1260	29-135	0-25

12.7 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 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. The following table lists the LCS recovery limits.

Compound	% Recovery QC Limits
AR1016	50-150

AR1260	50-150

12.8 Surrogate recoveries. The analyst must evaluate surrogate recovery data from individual samples against KAR-generated surrogate control limits. The following table lists an example of the limits. See the lab supervisor for current surrogate control limits.

Surrogate Recovery Table

Surrogate Compound	Matrix	Spike Amount	Surrogate Recovery Control Limit Range, %
TCMX	Aqueous	0.2 µg/L	7-84
DCB	Aqueous	0.2 µg/L	6-101
TCMX	Soil	200 µg/kg	22-95
DCB	Soil	200 µg/kg	24-122

These limits are updated every 6 months according to the procedures given in SW-846, method 8000.

12.9 See the appropriate KAR SOP for details regarding quality control procedures associated with sample preparation and analysis.

TABLE 1

SPECIFIC PCB CONGENERS IN AROCLORS

		Aroclor							
Congener	IUPAC number	1016	1221	1232	1242	1248	1254	1260	
Biphenyl	--		X						
2-CB	1	X	X	X	X				
23-DCB	5	X	X	X	X	X			
34-DCB	12	X		X	X	X			
244'-TCB	28*	X		X	X	X	X		
22'35'-TCB	44			X	X	X	X	X	
23'44'-TCB	66*					X	X	X	
233'4'6-PCB	110							X	
23'44'5-PCB	118*							X	X
22'44'55'-HCB	153							X	
22'344'5'-HCB	138							X	
22'344'55'-HpCB	180							X	
22'33'44'5-HpCB	170							X	

*Apparent co-elution of:
28 with 31 (2,4',5-trichlorobiphenyl)
66 with 95 (2,2',3,5',6-pentachlorobiphenyl)
118 with 149 (2,2',3,4',5',6-hexachlorobiphenyl)

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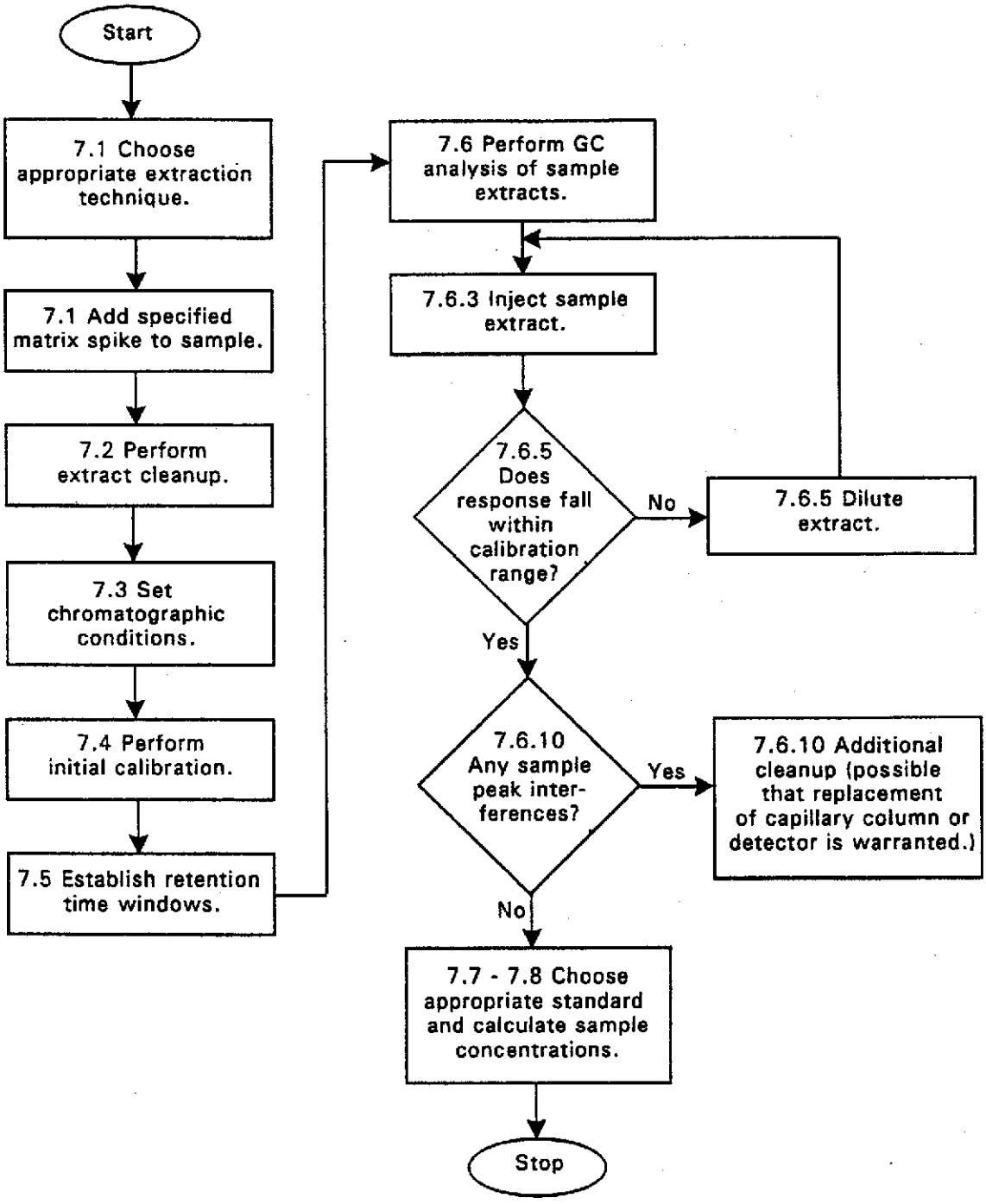
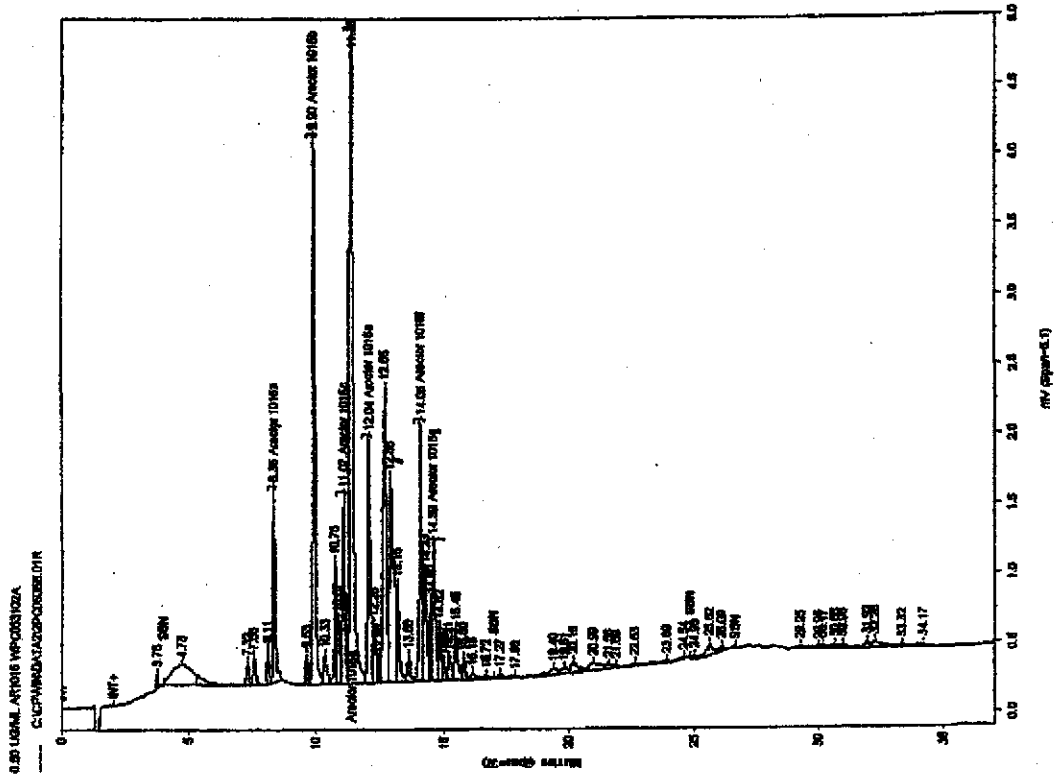
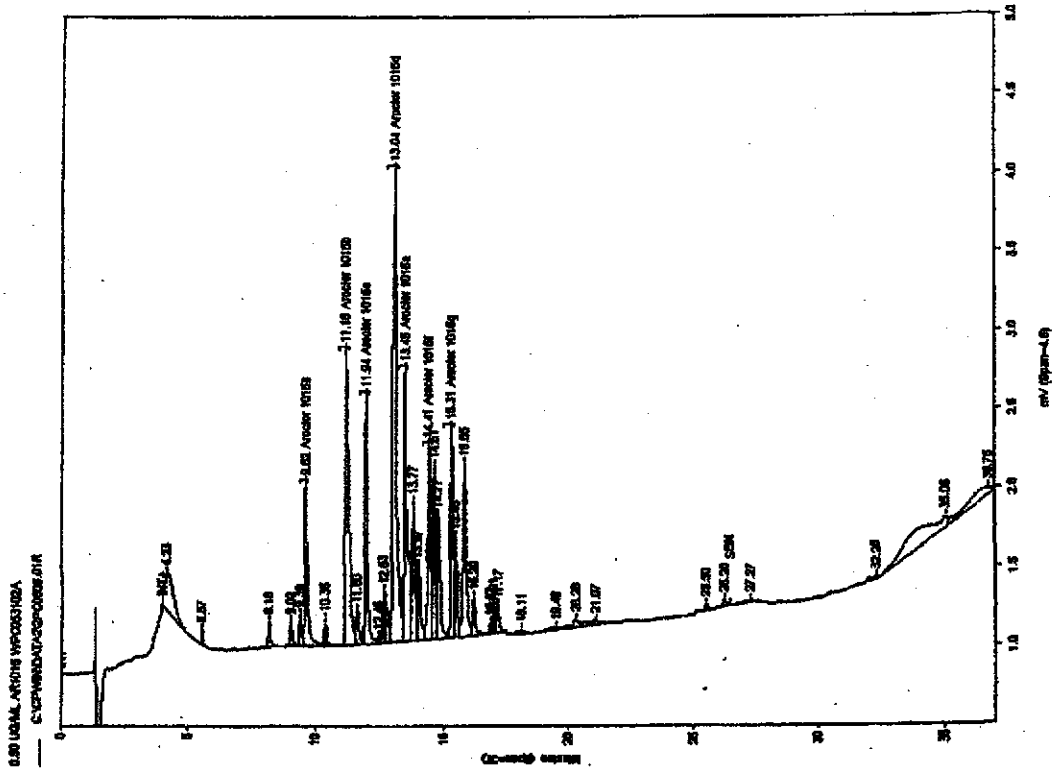
June 20, 2007

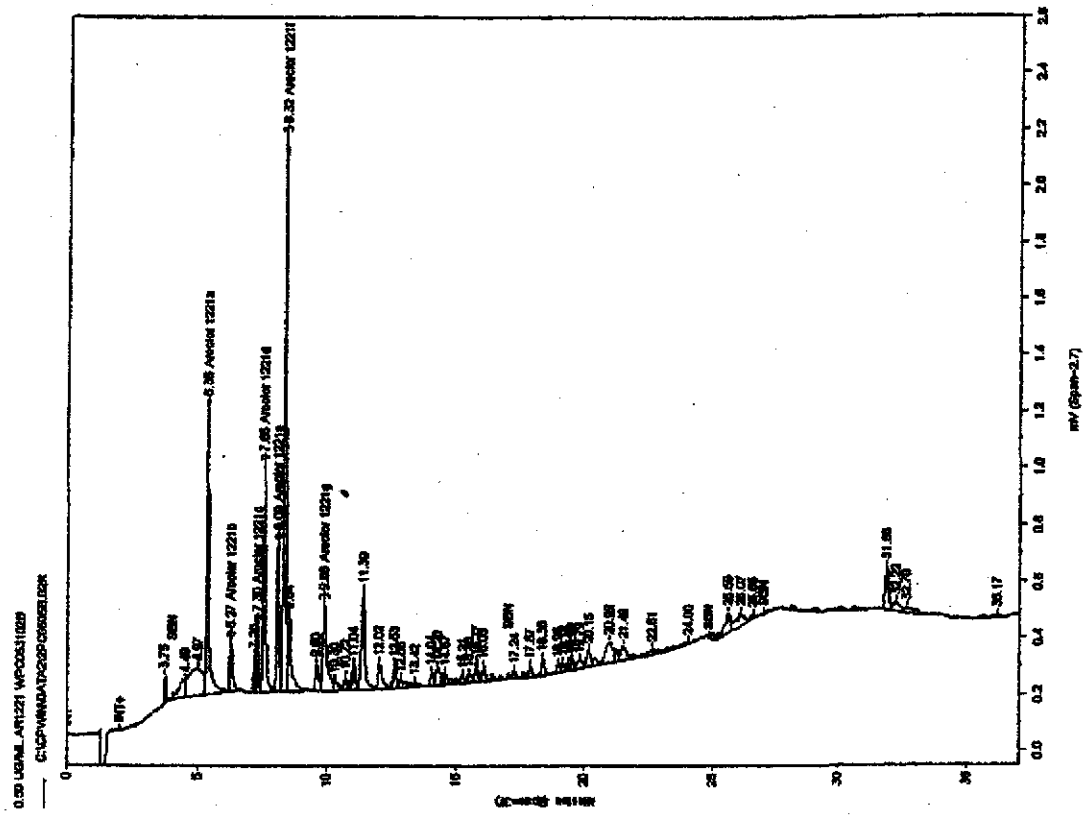
FLOW CHART

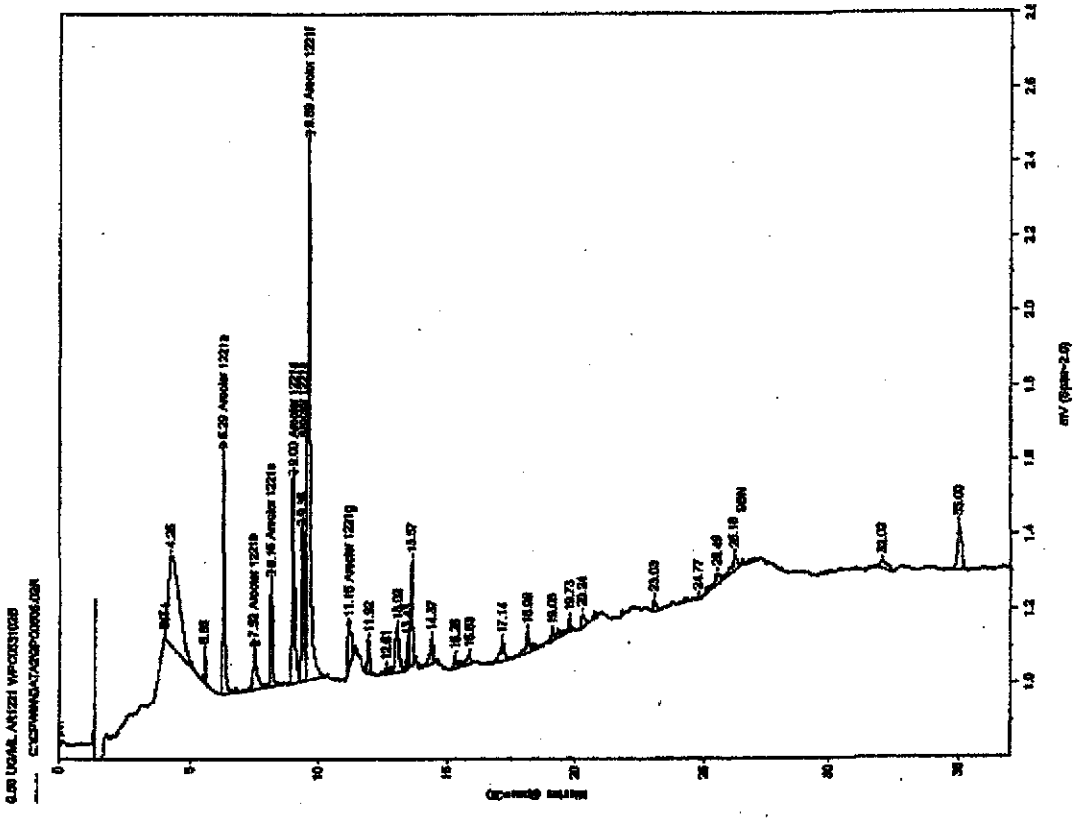
POLYCHLORINATED BIPHENYLS (PCBs) BY GAS CHROMATOGRAPHY

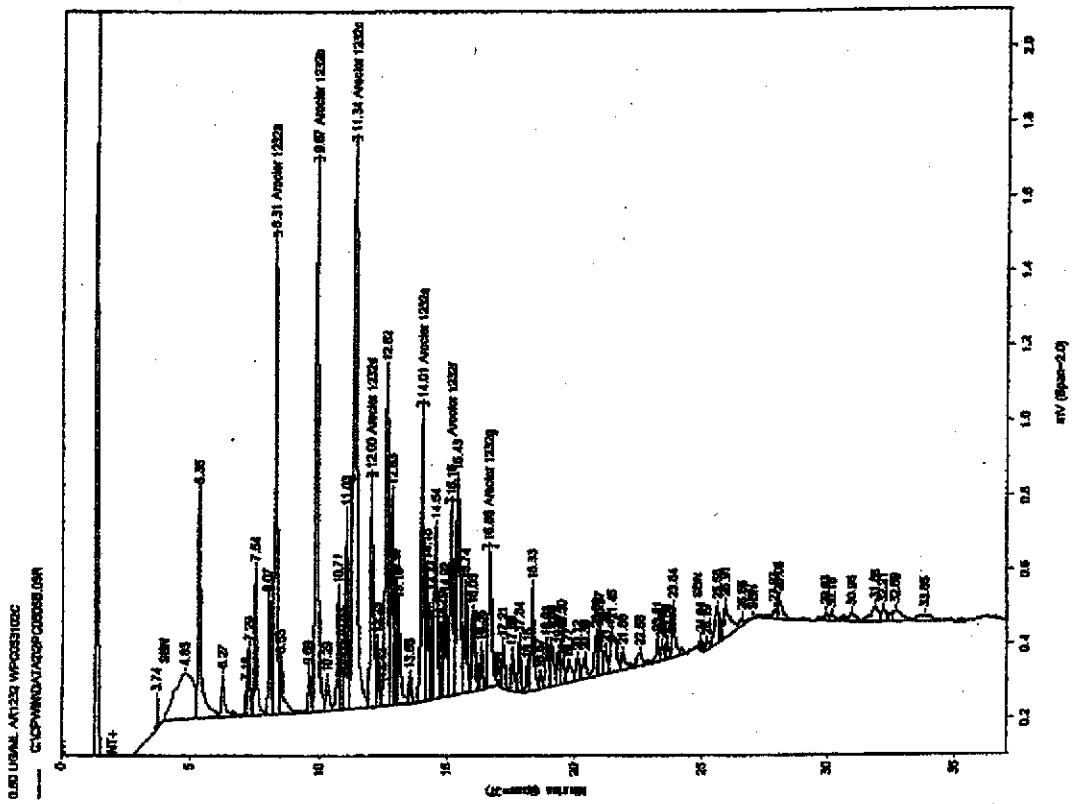


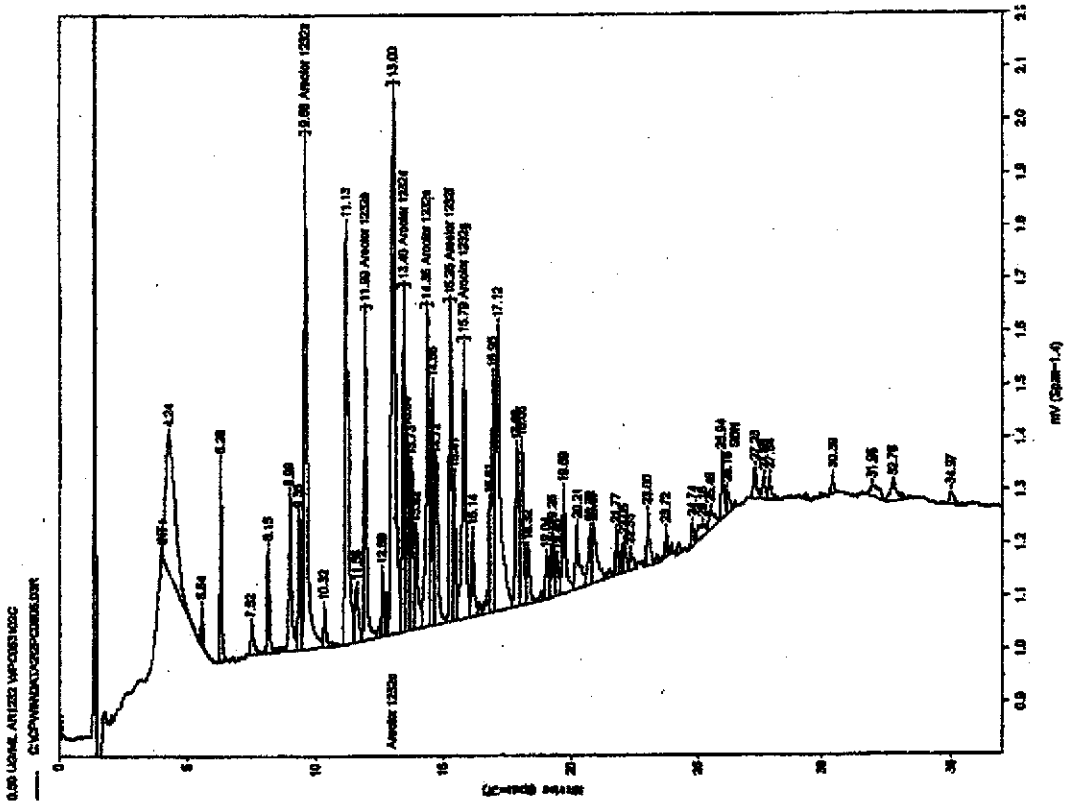


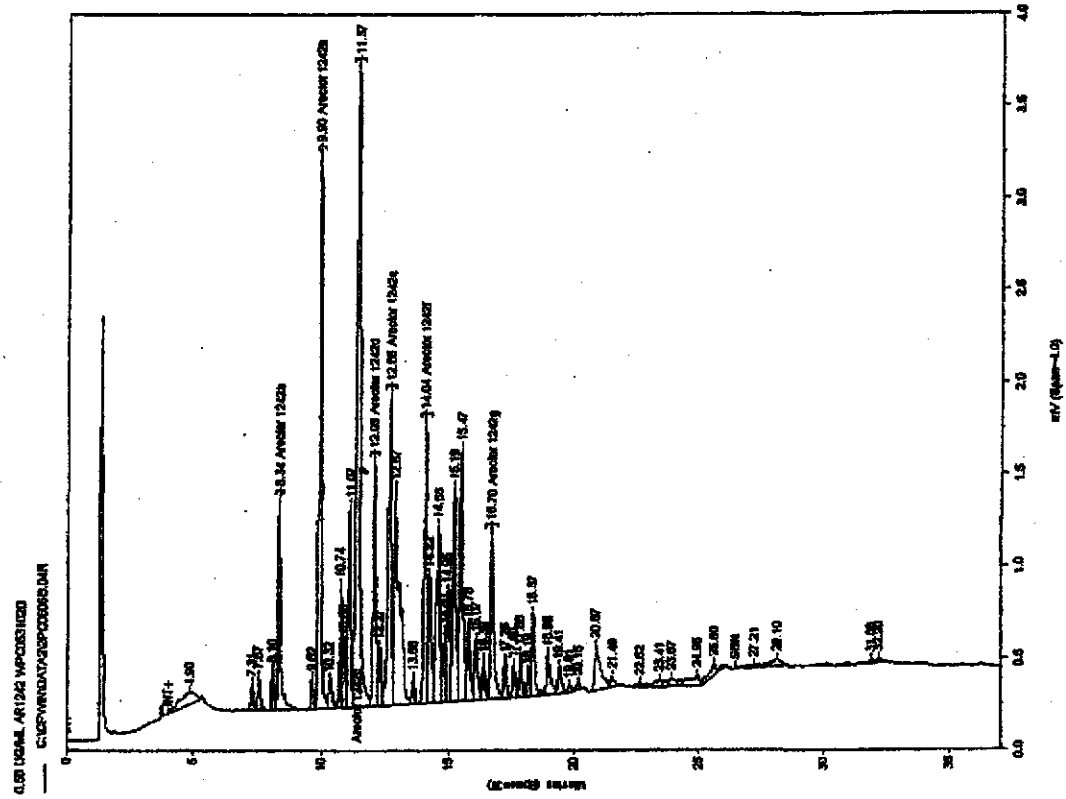


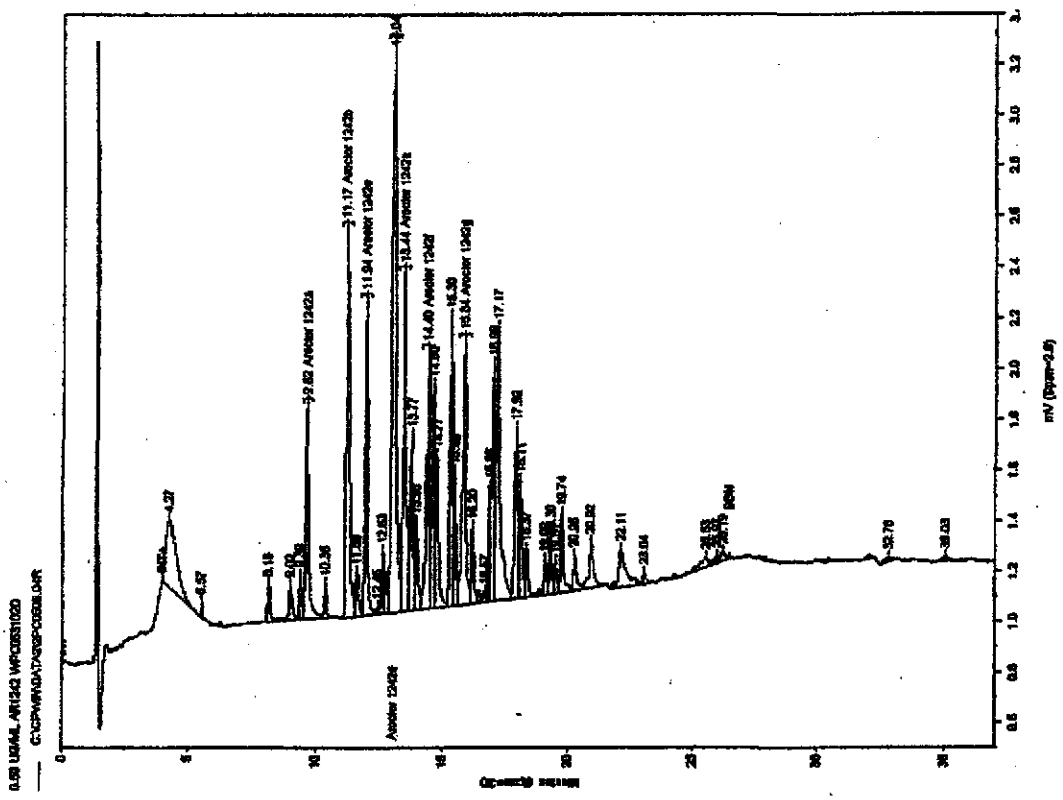


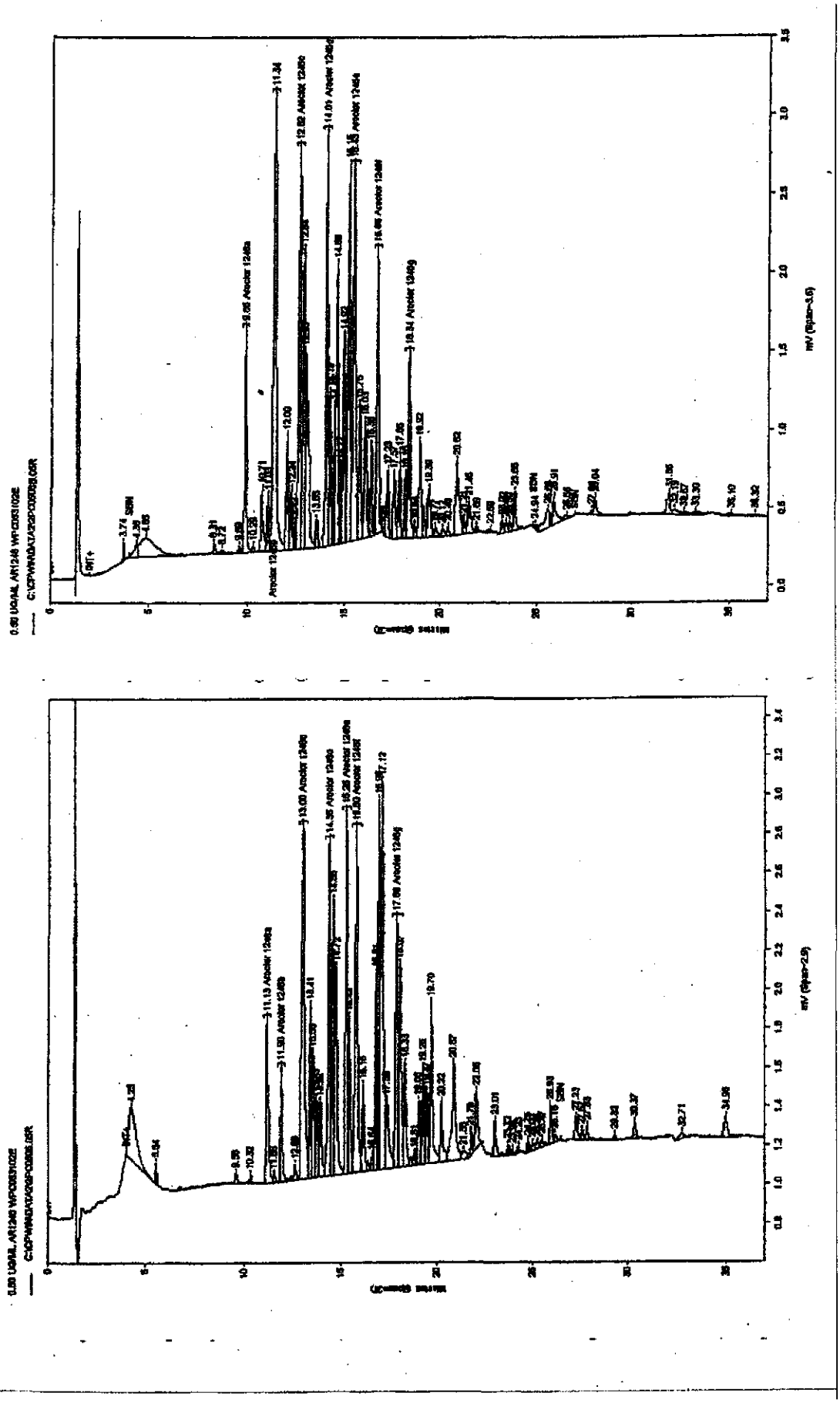


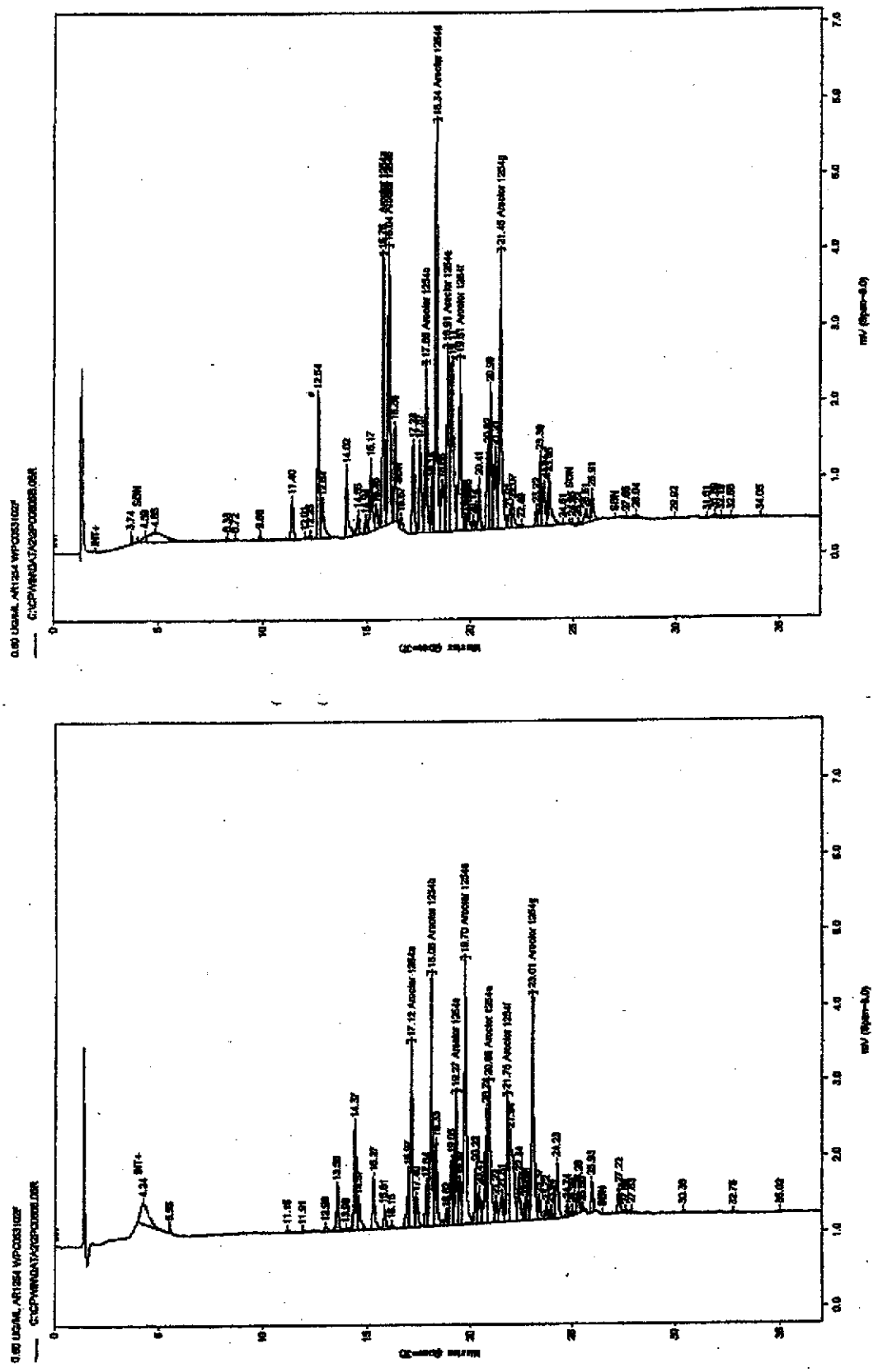


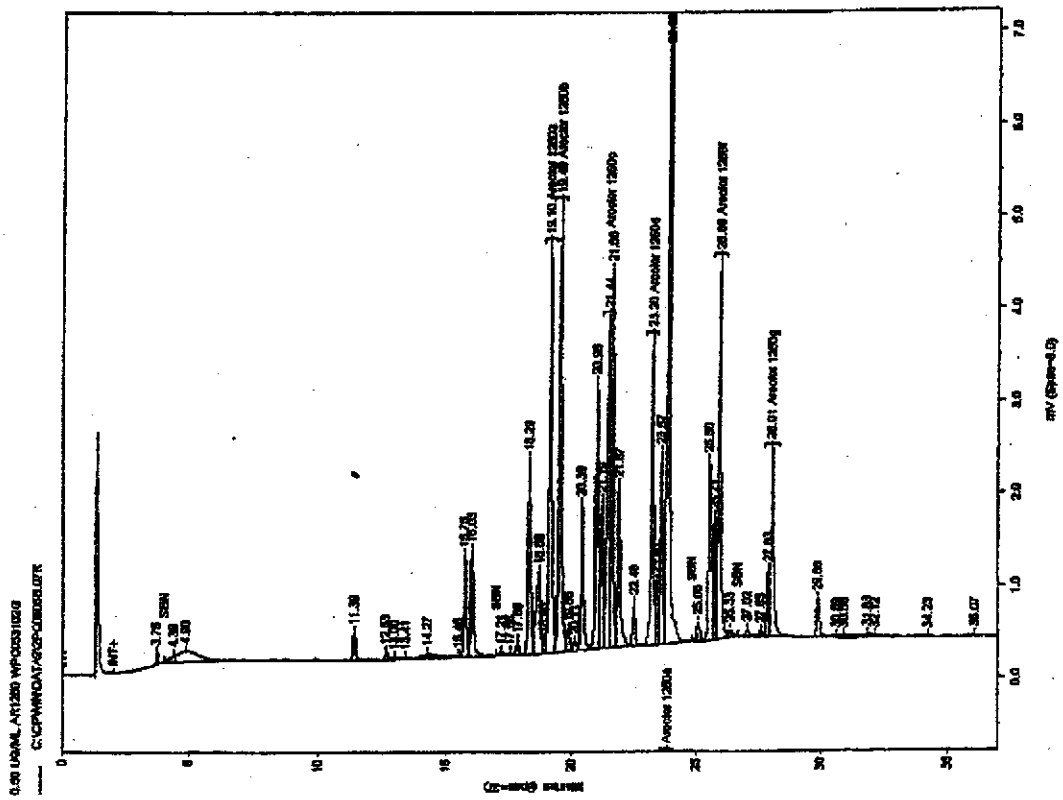


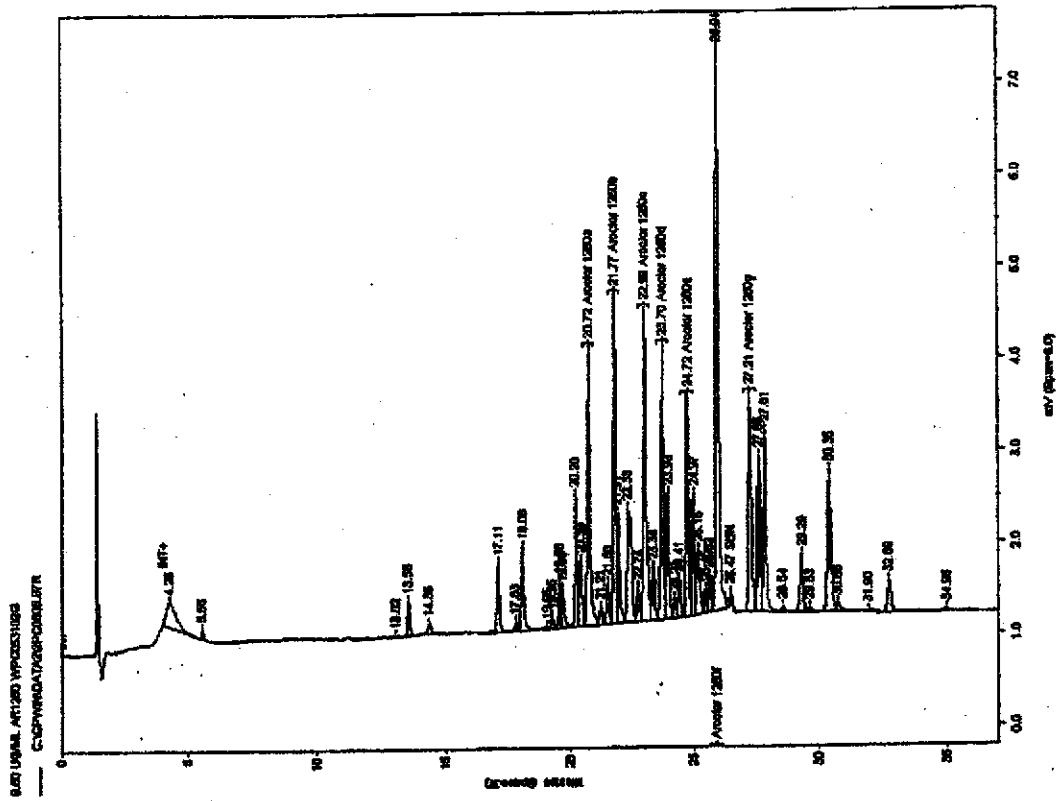


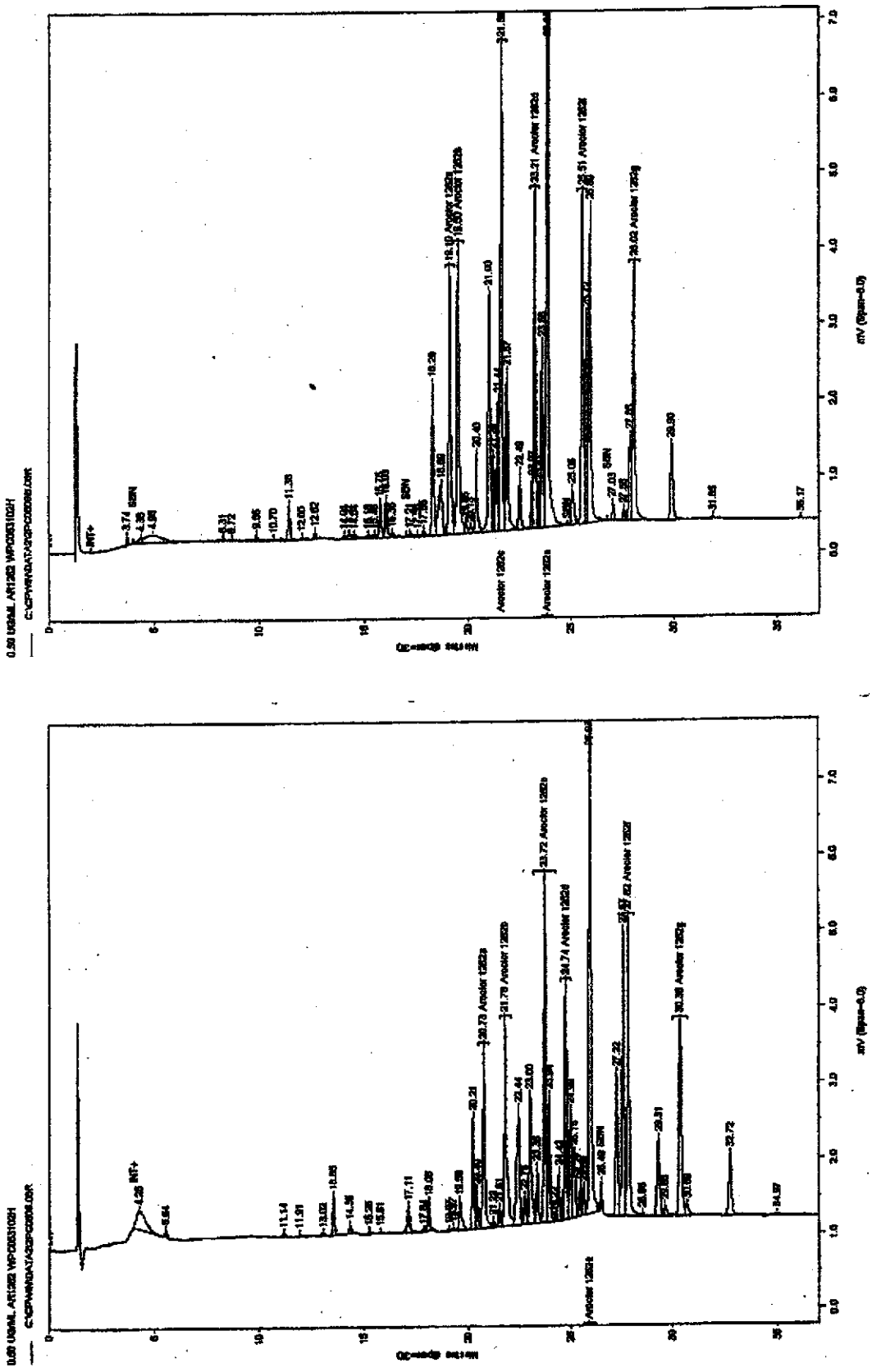


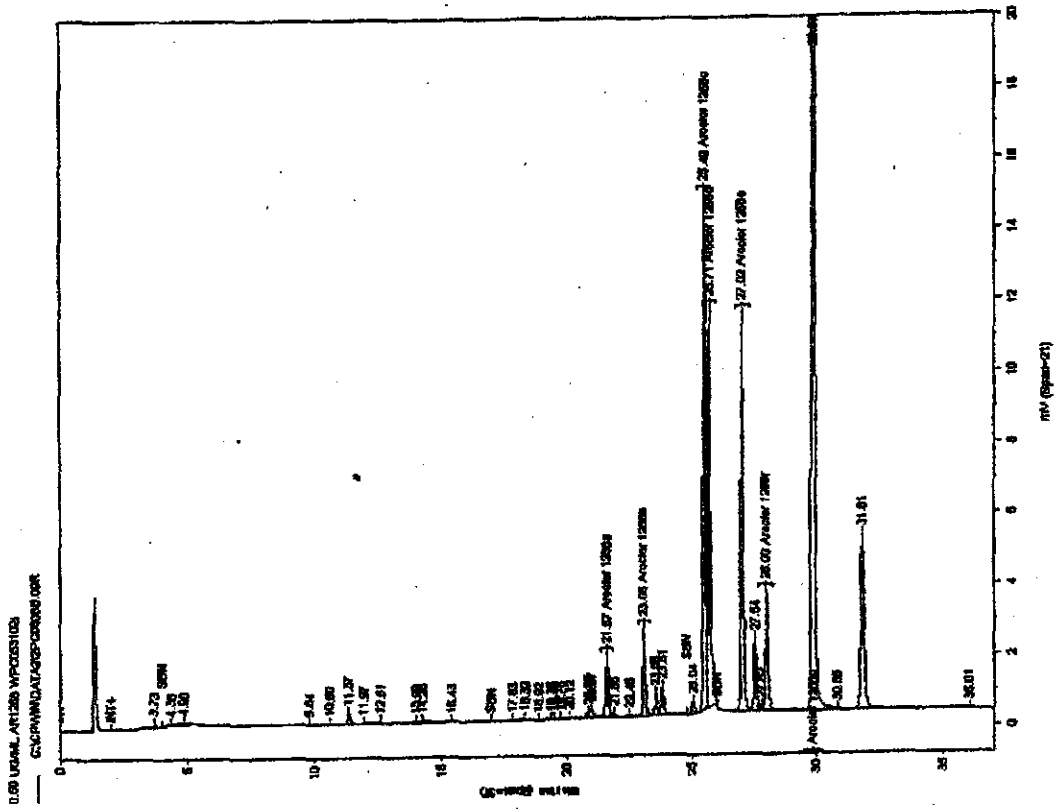


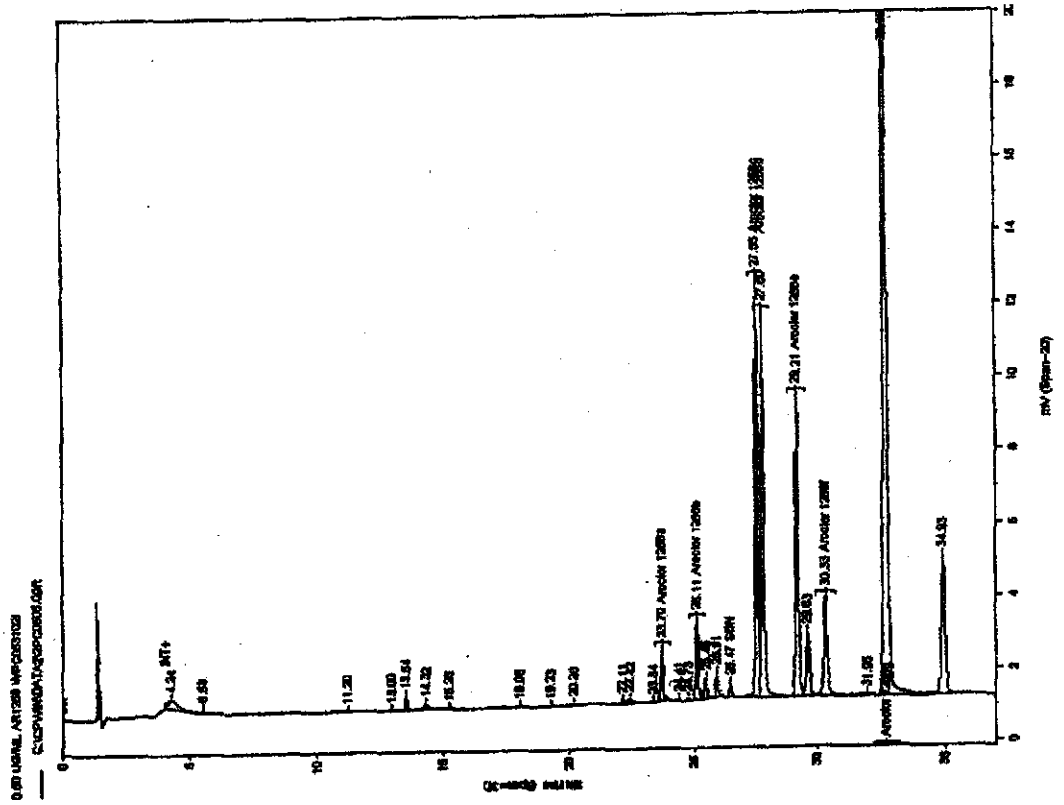












Signature Page

To the best of my knowledge all information contained in this document is complete and accurate.

Author: _____ Date: 6-14-07
Kent Lawrence

Supervisor: _____ Date: 6/14/07
Michael Jaeger

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Michael Jaeger

QA Approval: Linda Felcyn Date: June 14, 2007
Linda Felcyn

STANDARD OPERATING PROCEDURE

Turbovap Concentration

1.0 References:

1.1 "Turbovap Concentrator Operator's Manual," Zymark Corporation, 11/19/91.

1.2 EPA, "Test Methods for Evaluating Solid Wastes," SW-846, 3rd Edition, Update III, Method 3500B: Organic Extraction and Sample Preparation, U.S. EPA Office of Solid Waste and Emergency Response, Washington DC., December, 1996.

2.0 Application:

2.1 This procedure is a method for concentration of solvent extracts produced from separatory funnel extraction SOP (KO3510), solid phase extraction SOP (KO3535), and accelerated solvent extraction SOP (KO3545).

3.0 Summary of Method:

3.1 The Turbovap (TV) concentration workstation provides automated sample evaporation using a gas vortex shearing action to maintain high evaporation rates. A "helical flow," created by the stream of nitrogen directed into each sample, sets up a vortexing action that provides for sample homogeneity and continuous rinsing of the tube wall.

4.0 Safety:

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, dichloromethane and PCBs are suspected human carcinogens. Solvents and sample extracts should be treated as potential health hazards and exposure to them should be minimized. A reference file of Material Safety Data Sheets (MSDS) is available to all employees and the MSDS for each chemical used should be read prior to use. All solvents must be used in a fume hood to avoid inhaling the vapor. The hood must be operated at a safe face velocity as monitored by the velocity alarm on the hood. Never operate the turbovap units without the TV exhaust hoses placed in a fume hood. Gloves and safety glasses must be worn to avoid exposure through the skin and eyes from all reagents used. A labcoat is also recommended. Cracked, chipped or broken glassware must not be used.

Caution: Do not concentrate ethyl ether or MTBE to dryness as explosion may result.

5.0 Apparatus, Materials and Reagents:

- Turbovaps (TV) #1, #2, #3, #4; all Zymark TV II models
- 300 mL Turbovap concentration tubes with 1.0 mL calibration marks
- Pyrex powder funnels
- Glass wool, silanized
- Sodium sulfate, granular, 10-60 mesh, pesticide residue grade
- Methylene chloride, pesticide residue grade
- Hexane, pesticide residue grade
- Acetone, pesticide residue grade
- Turbovap tube racks
- Autosampler vials, 1.5 mL with PFTE-lined crimp caps
- Crimper
- Disposable Pasteur pipettes, 9"
- Pipette bulb
- Sharpe® marker

6.0 Procedure:

Organization and Set-up

6.1 For each sample to be concentrated, prepare a TV tube, a powder funnel with a small plug of glass wool and approximately 15 grams of sodium sulfate, and an autosampler vial with cap.

6.2 Rinse the sodium sulfate/funnels with approximately 10-20 mL of methylene chloride and allow to air dry in hood. Rinse the TV tubes three times each with methylene chloride and allow to dry upside down in racks in hood.

6.3 Using a Sharpe® marker, label enough pre-calibrated autosampler vials for all the sample extracts being concentrated with the appropriate sample number and analysis to be performed. There should be enough space on the LFB, LMB, MS and MSD labels to also include the extraction date.

6.4 TV Water bath level - Fill with deionized water so that the water bath level will be higher than the solvent level in the TV tubes. (In addition, water in bath must be changed monthly. Refer to maintenance section for procedure).

6.5 Nitrogen supply - Check nitrogen supply and turn on the main N_2 value in the gas storage room. Turn the N_2 control knob on the hood approximately ¼ turn counterclockwise (on).

6.6 Turn on power to TV workstations.

6.7 Set TV to the following settings:

CONTROL BOARD SETTINGS

Endpoint select - sensor
Bath temp - 40° C to 50° C
Endpoint time - 0

6.8 For most situations, Turbovap workstations are set above the boiling point of the solvent being used because the nitrogen has a cooling effect on the surface of the solvent. See Table I for specific solvent temperatures and pressures.

Table I. Turbovap Temperature Pressure Settings

Solvent	Temperature	Pressure start	Pressure end
Methylene chloride	**40^{0}C**	**15psi**	**20psi**
Ethyl ether	**40^{0}C**	**15psi**	**20psi**
Hexane	**50^{0}C**	**15psi**	**20psi**
Methanol	**50^{0}C**	**15psi**	**20psi**

Concentration

6.9 Dry each sample extract by passing it through the solvent-rinsed sodium sulfate/funnel and into the TV tube. Rinse the sample bottle two times with methylene chloride, decanting it into the funnel/TV tube after each rinse. Thoroughly rinse the sodium sulfate/glass wool with methylene chloride into the TV tube so as to pass any residual sample components into the TV tube. The sample extracts are now ready for concentration.

6.10 After the water bath has warmed to the proper temperature, carefully insert TV tubes into position. (**WARNING:** The reservoir tips of the TV tubes are very fragile, handle with caution. If a TV tube breaks and the $MeCl_2$ extract gets into water bath, remove all TV tubes immediately. Drain and clean water bath immediately.)

NOTE: Ensure proper spacing of samples in the water bath with known high concentrations of target analytes to avoid possible contamination.

6.11 When all tubes are in position, lower TV cover.

6.12 Turn N_2 supply on at the hood.

6.13 Press start control for each position in use. Adjust N_2 pressure using the pressure regulator located on the lower left side of TV so that a vortexing action is created in each tube.

Do not raise the N_2 pressure too high, in order to prevent splashing. Ideally, pressure should be between 10-20 psi; however, when extracts contain greater than 200 mL, a pressure less than 8 psi (~ 4-6 psi) may be necessary. As sample extracts concentrate, check intermittently and adjust N_2 pressure as high as possible (up to 20 psi) to maintain the vortexing action and to concentrate the extract as quickly as possible in order to avoid analyte loss. Concentration of 200-300 mL of methylene chloride should take no longer that 15-20 minutes.

NOTE: Operator must attend samples when volume reaches 10 mL to ensure samples achieve appropriate final volume. TV sensors are prone to failure.

6.14 When sample extracts have concentrated to ~ 2 mL, the following procedures may be performed if necessary.

6.14.1 Solvent exchange

6.14.1.1 PCB/Pesticide extracts require the addition of ~ 30 mL hexane.

6.14.1.2 Sonication extracts require the addition of 30 mL $MeCl_2$.

6.14.1.3 Formaldehyde extracts require the addition of 30 mL methanol.

6.14.1.4 Benzidines by 605 extracts require the addition of 30 mL methanol.

6.14.1.5 Herbicide extracts require the addition of 30 mL MTBE.

In all cases of solvent exchange, the sample/solvent mixture should be hand-swirled in the TV tube after the exchanging solvent is added to facilitate mixing.

NOTE: If the original sample extract exceeded 200 mL, add additional amounts of sample extract which require concentration. This will always be the case for extracts from continuous liquid-liquid extraction. Use the same funnel as previously used to add sample extract to TV tube.

6.15 Concentrate sample extracts to no less than 0.5 mL. Do not concentrate sample extracts to dryness or irreversible loss of sample components may occur. If any extract goes to dryness, it must be re-extracted and/or flagged as estimated result. In all cases of extract loss or near loss (<0.5 mL), supervision must be notified immediately. CONCENTRATION OF ETHYL ETHER OR MTBE TO DRYNESS MAY RESULT IN EXPLOSION. Remove TV tubes from TV.

Final Extract Transfer

6.16 Hold the Turbovap tube within the line of sight.

6.17 Using a 9" disposable Pasteur pipette, add enough fresh solvent to bring the sample to the 1mL mark by rinsing down the side walls of the tube in a swirling motion at the point just above where the tube narrows.

6.18 Take the entire 1 mL sample into the pipette and rinse the same lower portion of the Turbovap tube.

6.19 Repeat the above technique two more times. At this point, the extract will have evaporated to about 0.5 mL.

6.20 Repeat step 6.16 with fresh solvent and bring to the final 1 mL mark.

6.21 Using the same pipette, the 1 mL sample extract is now ready for transfer to an autosampler vial. (If 1 mL is not the final endpoint, perform a quantitative transfer. See Table II for appropriate final volumes.)

Table II. Final volumes and solvents for Turbovap concentration

Method	Aqueous	Soil	Sludge (<5% solids)
EPA 8270C Acid, base/neutral extractables by GC/MS	Final volume = 1.0 mL solvent = $MeCl_2$	Final volume = 1.0 mL solvent = $MeCl_2$	Final volume = 1.0 mL solvent = $MeCl_2$
EPA 8081/8082 Pesticides and PCBs by GC/ECD	Final volume = 1.0 mL solvent = Hexane	Final volume = 10.0 mL solvent = Hexane	Final volume = 5.0 mL solvent = Hexane
Aldehydes by EPA 1776/8315	Final Volume = 10.0 mL solvent = MeOH	Final Volume = 10.0 mL solvent = MeOH	Final Volume = 10.0 mL solvent = MeOH
Herbicides by EPA 8151/515.1	Final Volume = 5.0 mL Solvent - MTBE	Final Volume = 5.0 mL Solvent - MTBE	Final Volume = 5.0 mL Solvent - MTBE
Benzidines by 605	Final Volume = 5.0 mL Solvent = MeOH	Final Volume = 5.0 mL Solvent = MeOH	Final Volume = 5.0 mL Solvent = MeOH

6.22 Sample extract is now ready for additional cleanup or analysis. Refer to appropriate SOP.

Shutting down

6.23 Store TV with cover in the up position to prevent moisture from accumulating on the inside cover.

6.24 Replace the plastic caps which cover the TV tube positions in the water bath.

6.25 Turn N_2 regulators to the lowest flow position.

6.26 Turn off N_2 supply at the hood and at the N_2 source in the gas storage room.

6.27 Turn off the power to the TV units.

6.28 Store sample extracts in designated vial file storage unit and record the final volume and file location in appropriate logbook. Samples may be stored frozen (-5° C to -25° C) for up to 40 days before analysis.

7.0 Maintenance:

7.1 **Monthly** - Since turbid water may affect sensor operation, the water in the water bath must be replaced monthly. Using the water bath siphon pump, empty the water from the bath. Fill the bath with deionized water until the water level is approximately 1 inch from the top. Add 15 drops of Clear Bath, a biological growth retardant. Use the bubble dislodger to dislodge bubbles from the sensors. Occasionally, remove the rack from the bath after unscrewing the center bolt and clean the bath walls. Record all maintenance in the logbook corresponding to the appropriate turbovap.

7.2 **Quarterly** - Perform cell diagnostics per the manual instructions to test sensors, switches, lights, gas pressure and valves. Replace sensors and/or switches when necessary. Record all maintenance in the logbook corresponding to the appropriate turbovap.

8.0 QA/QC:

8.1 Management is responsible for bi-annual review of turbovap maintenance logbooks in order to assure proper and timely maintenance is being performed.

8.2 In all instances of sample loss due to TV tube breakage or evaporation to dryness, management must be notified immediately.

Signature Page

To the best of my knowledge all information contained in this document is complete and accurate.

Author: _____ Date: 7-28-05
Kent Lawrence

Supervisor: _____ Date: 7/29/05
Michael Jaeger

Laboratory Director: _____ Date: 7/29/05
Michael Jaeger

QA Approval: _____ Date: 1 Aug 05
Pollyann Nee

STANDARD OPERATING PROCEDURE

Separatory Funnel Extraction by EPA Method 3510C

1. Reference:

1.1 Separatory Funnel Liquid-Liquid Extraction, Method 3510C EPA SW-846.
1.2 "Sulfuric Acid/Permanganate Cleanup by EPA Method 3665A," KAR Laboratories' SOP KO3665A.
1.3 "Florisil Cleanup by EPA Method 3620B," KAR Laboratories' SOP KO3620B.
1.4 "Sulfur Cleanup by EPA Method 3660A," KAR Laboratories' SOP KO3660A.
1.5 "Turbovap Concentration," KAR Laboratories' SOP KO3500B.

2. Application:

This procedure is used for the solvent extraction of compounds from aqueous samples to be analyzed for semi-volatile organic compounds by EPA Methods 8270, 8081, 8082, or 680.

3. Interferences:

3.1 Phthalate esters, found in many plastics, are a major source of contamination. Therefore, it is crucial that samples and reagents contact glass or Teflon **only**. In particular, the following precautions must be taken:
- Plastics may not be used in any part of the procedure (including plastic pipets and squirt bottles).
- Gloves or glove powder must not come in contact with any surface in which the sample or extract is exposed.

3.2 PCB samples may be subjected to cleanup methods to remove interferences if necessary. (See SOPs KO3620B, KO3660A and KO3665A.)

4. Safety:

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, $MeCl_2$ and PCBs are suspected human carcinogens. Solvents, samples, reagents and spiking and surrogate solutions should be treated as potential health hazards and exposure to them should be minimized. A reference file of Material Safety Data Sheets (MSDS) is available to all employees and the MSDS for each chemical used should be read prior to use. $MeCl_2$ must be used in a fume hood to avoid inhaling the vapor. The hood must be operated by a safe face velocity

as monitored by the velocity alarm on the hood. When dumping aqueous samples, you should use the sinks located in the hoods. SAMPLES MUST NOT BE DUMPED UNTIL THE PROJECT HAS BEEN REPORTED OR 14 DAYS, WHICHEVER IS LONGER. Gloves and safety glasses must be worn to avoid exposure through the skin and eyes from all reagents used. A labcoat is also recommended. Cracked, chipped or broken glassware **must not** be used. Special attention must be paid to separatory funnels. Star cracks in the separatory funnel may cause the separatory funnel to explode when pressure is created inside during shaking.

4.2 Due to the hazardous nature of PCBs, extreme measures must be taken to ensure proper disposal of all PCB contaminated samples and/or reagents. ANY PCB RESULT $\geq$ 50 PPM MUST IMMEDIATELY BE REPORTED TO THE DEPARTMENT SUPERVISOR OR DESIGNEE. The supervisor is then responsible for collecting all affected samples/extracts and forwarding them to the waste manager for proper disposal. SAMPLES CONTAINING PCBS $\geq$ 50 PPM MUST NEVER BE POURED DOWN THE DRAIN.

5. Apparatus, Materials and Reagents

2 L Separatory Funnels with stopcocks and stoppers
Thermoline Automated Separatory Funnel/Shaker
Mettler PM 5600 balance or equivalent
Graduated Cylinder, 500 mL
Bottles, amber 500 mL with teflon lined screw caps
Disposable Pasteur pipettes, 9”
Pipette bulb
Erlenmeyer Flasks, 1 L
pH paper
2 L tipping dispenser with 60 mL dispensing head
Glass wool
Glass stir rod
Centrifuge, Megafuge 1.0 or equivalent
Centrifuge Tubes, 500 mL
Methylene Chloride, pesticide residue grade
Carbon-Filtered Deionized Water (c-dH_20)

1:1 Sulfuric acid (H_2SO_4): Carbon-Filtered Deionized Water

Slowly add 250 mL of H_2SO_4 to 250 mL of c-dH_20 in a 1 L erlenmeyer flask. CAUTION: Solution will be very hot; allow to cool to room temperature before use. NEVER add water to acid! Store at room temperature for up to 1 year.

10 N Sodium Hydroxide (NaOH)

Weigh 200 g of NaOH pellets into a 1 L erlenmeyer flask. Add 500 mL of c-dH_20 and stir using a glass rod to dissolve the pellets. CAUTION: Solution will be hot; allow to cool to room temperature before using. Store at room temperature for up to 1 year.

6. Procedure:

Organization and Set-up

6.1 Remove the samples to be extracted from the refrigerator. This initial set of samples and corresponding QC samples are the QC batch. Each QC batch must include a laboratory method blank (LMB), laboratory fortified blank (LFB), matrix spike (MS) and matrix spike duplicate (MSD) of a sample.

6.1.1 Sample volume is approximately 1 L for most samples. Low level PCB samples require 2 L of sample.

6.1.2 The LMB and LFB are approximately 1L of carbon-filtered deionized water (C-dH_2O). The LFB is a fortified spike of reagent water. Both the LMB and LFB are treated in the same manner as the samples.

6.1.3 One set of spikes, a MS and MSD, is extracted for each QC batch not exceeding 20 samples. Select a sample with 3 L of sample available. Label one sample container MS and one MSD, leaving one container for the unspiked sample.

If only 2 L are available, split one liter of the sample into two portions using a 500 mL graduated cylinder to measure their volumes. Record the sample volumes for the spikes in the appropriate extraction lab notebook.

If none of the samples have more than 1 L of sample available, extract a LFB and LFB-duplicate instead of the MS and MSD.

6.2 Remove the appropriate surrogate(s) and spike(s) from the freezer as determined from the "Surrogate and Spike Chart."

6.3 Rinse one 2 L separatory funnel and stopper with $MeCl_2$ for each sample after checking the separatory funnel for star cracks. Rinse one Wheaton 500 mL amber bottle with $MeCl_2$ for each of the following samples which does not have an original sample container: LMB, LFB, MS, MSD. If performing a complete ABN extraction, rinse one additional 500 mL amber bottle with $MeCl_2$ for all samples and QCs.

6.4 Label the separatory funnels and amber bottles with the sample identification. Include the date and extraction type on all sample and amber containers.

Sample preparation

6.5 Check the separatory funnels to make sure the stopcocks are closed. Take the sample to the balance and tare the entire sample, jar and lid. Pour the sample into the appropriately labeled separatory funnel. Be careful not to touch inner lip of sep funnel with gloves. Immediately return to the balance and weigh the now empty sample jar. Record the negative weight reading from the balance on the bench sheet as mLs of sample. Repeat for all samples.

6.6 Check pH by dipping a disposable pipet into sample and distributing sample onto a piece of 1-14 indicator paper. Compare colors to pH chart. Repeat, using a new pipette for each sample.

6.7 Add necessary surrogate and spikes (refer to "Surrogate and Spike Chart).

6.8 Adjust pH if necessary. After adjustment, check the pH in the same manner as section 6.6 and record the pH measured of each sample on the extraction batch sheet.

6.8.1 Acid (A) extraction - adjust pH to less than or equal to 2 with (1:1) H_2SO_4.

6.8.2 Base (B) extraction - adjust pH to greater than or equal to 12 with 10 N NaOH.

6.8.3 Neutral (N) extraction (including PCB extraction)
pH should be between 5 and 9. If pH is not in this range, adjust sample accordingly with (1:1) H_2SO_4 or 10 N NaOH.

NOTE: Each sample must have a LMB associated with it that is treated in the same manner as the sample. For example, if the pH of a sample is changed, the LMB must have the same amount of acid or base added to it.

6.8.4 ABN extraction -. Change pH to less than or equal to 2 and follow complete extraction procedure for A extraction. Adjust pH to greater than or equal to 12 and follow complete extraction procedure for B extraction. Always do A extraction before B extraction as is stated in EPA 3510C.

Extraction

6.9 Add 60 mL of $MeCl_2$ to sample container from tipping dispenser. Cap container and gently swirl the sample bottle a few times in order to rinse the container sides with solvent. Carefully pour the $MeCl_2$ into the separatory funnel with the associated sample.

6.9.1 Manual Method: After replacing stopper, invert sep funnel and open stopcock to relieve pressure. Open and close the stopcock repeatedly while shaking until pressure build-up is no longer observed. Shake sample for 2 minutes.

6.9.2 Automated Method: Loosely cap the automated separatory funnels and turn the unit on low speed (1-3 setting), gradually increasing the speed to 14-18 (setting) while simultaneously tightening down the self-venting caps. Let the unit(s) shake for 3 minutes.

6.10 Let aqueous and $MeCl_2$ layers separate for a minimum of 10 minutes. Drain $MeCl_2$ extract into sample container. (If not available, collect extract in Wheaton 500 mL amber bottle). If layers do not separate, manual techniques for separation may be necessary. The experience of the technician will be crucial in choosing one of the following techniques. If necessary, ask for direction from the supervisor.

6.10.1 Use a solvent rinsed stirring rod to break up the emulsion in the separatory funnel.

6.10.2 Filter extract through solvent rinsed glass wool.

6.10.3 Transfer extract to a centrifuge tube. Centrifuge for 5 minutes at approximately 1300 rpm. Transfer contents of the centrifuge tube back to the separatory funnel and drain $MeCl_2$ extract as in 6.9. NOTE: It may be impractical to deal with emulsions during the extraction steps. In these instances, the emulsion may be separated before sample concentration.

6.11 Repeat sections 6.9 and 6.10 two more times with 60 mL aliquots of $MeCl_2$ added directly to sep funnel. Combine $MeCl_2$ extracts in sample container.

6.12 Sample extracts may be stored in Teflon-lined screw cap amber bottles at 4^0 C for up to 10 days before concentration.

Concentration

6.13 Proceed to turbovap concentration SOP KO3500B.

7. QA/QC

7.1 Extraction Batch: The extraction batch is defined as all samples extracted by this method during a single day and must include a minimum of one LMB per extraction batch not to exceed 20 samples.

7.2 QC Batch: The QC batch is defined as an extraction batch which also contains an LFB, MS, and MSD. The QC batch may contain as many extraction batches as necessary up to 20 samples or 30 days, whichever comes first.

7.3 Any reagent blanks, matrix spikes, laboratory fortified samples or replicate samples should be subjected to exactly the same analytical procedures as those used on actual samples.

Signature Page

To the best of my knowledge all information contained in this document is complete and accurate.

Author: _____ Date: 8-24-05
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Supervisor: _____ Date: 8/25/05
Michael Jaeger

Laboratory Director: _____ Date: 8/25/05
Michael Jaeger

QA Approval: _____ Date: 25-Aug-05
Pollyann Nee

STANDARD OPERATING PROCEDURE

Accelerated Solvent Extraction **(3545)**

1. References:

1.1 U.S. EPA's "Test Methods for Evaluating Solid Wastes," SW-846, 3rd Edition, Update III, December, 1996.

1.2 "Analysis of PCB by EPA Method 8082," KAR Laboratories, Inc. SOP KP8082.

1.3 "The Gravimetric Determination of Moisture and Total Solids at 103-105° C," KAR Laboratories, Inc. SOP KQ462.

1.4 "Herbicide Sample Prep by EPA Method 8151A," KAR Laboratories, Inc. SOP KP8151A-pre.

2. Application:

2.1 This procedure is used for extracting water insoluble or slightly water soluble semivolatile organic compounds from soils, clays, sediments, sludges and waste solids. The method uses elevated temperature (90° - 100° C) and pressure (1500 - 2000 psi) to achieve analyte recoveries equivalent to those from soxhlet extraction, using less solvent and taking significantly less time than the soxhlet procedure. This procedure was developed and validated on a commercially-available, automated extraction system.

This method is applicable to the extraction of semivolatile organic compounds, including, organophosphorus pesticides, organochlorine pesticides, total petroleum hydrocarbons (TPH), and PCBs, which may then be analyzed by a variety of chromatographic procedures.

This method has been validated for solid matrices containing 250 to 12,500 µg/kg of semivolatile organic compounds, 250 to 2500 µg/kg of organophosphorus pesticides, 5 to 250 µg/kg of organochlorine pesticides, and 1 to 1400 µg/kg of PCBs.

3. Summary of Method:

3.1 Approximately 10 g of sample are mixed with approximately 5 g of diatomaceous earth (or Hyromatrix®), and loaded into a 30 mL extraction cell.

Surrogates/spikes are added. The sample is equilibrated for 5 minutes and extracted for 6 minutes using the appropriate solvent at elevated temperature (95° - 100° C) and elevated pressure (1800 - 2000 psi).

3.2 The extraction cell is allowed to cool to room temperature and the solvent is collected in a 60 mL glass vial.

3.3 The extract may be exchanged into a solvent compatible with the cleanup or determinative step being employed.

4. Interferences:

4.1 Phthalate esters found in many plastics are a major source of contamination. Therefore it is crucial that samples and reagent contact glass or Teflon only. In particular, the following precautions must be taken:

4.1.1 Plastics must not be used in any part of the procedure (including plastic pipets and squirt bottles).

4.1.2 Gloves or glove powder must not come in contact with any surface in which the sample or extract is exposed.

4.1.3 PCB samples may be subjected to cleanup methods to remove interferences if necessary. (See SOP KO8082 for PCB Cleanup Methods).

5. Apparatus & Materials:

5.1 Automated Accelerated Solvent Extractor (ASE) - Dionex Accelerated Solvent Extractor with extraction cells.

5.2 Vials for collection of extracts - 60 mL, pre-cleaned, open top screw-cap with PTFE-lined silicone septum.

5.3 Analytical Balance: Mettler PM 5600 or equivalent
Glass Beakers: 250 mL and 150 mL Pyrex®
7 mL clear vials with PTFE lined caps (Supelco #27150-0)
Turbo Vap tubes: 300 mL
Separatory funnels with glass stoppers: 250 mL
Amber bottles with screw caps: 250 mL
Wooden spatulas: Fisher Brand tongue depressors
Glass fiber filters: Dionex 1.91 cm GF/B
Pyrex® powder funnels
Syringes: 1 mL, 100 µL
Metal spatulas, black Sharpe® black felt tip marker
pH strips, 1-14 range

6. Reagents:

6.1 Acetone - pesticide residue grade
Ethyl Ether - pesticide residue grade
Methanol - pesticide residue grade
Methylene chloride - pesticide residue grade
Nitrogen gas, high purity for purging extraction cell
Diatomaceous earth - Celite® brand pesticide residue grade certified, for drying samples
Ottawa Sand - purified grade
Hydromatrix® - Varian #0019-8003
Silicic acid - 100 mesh
37% Potassium Hydroxide in deionized water (stored at 4° - 10° C)
Sulfuric Acid - 1:3 with deionized water (stored at 4° - 10° C)
Acidifed Sodium Sulfate

7. Safety:

7.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, $MeCl_2$ and PCBs are suspected human carcinogens. Solvents, samples, reagents and spiking and surrogate solutions should be treated as potential health hazards and exposure to them should be minimized. A reference file of Material Safety Data Sheets (MSDS) is available to all employees and the MSDS for each chemical used should be read prior to use. All solvents must be used in a fume hood to avoid inhaling the vapors. The hood must be operated at a safe face velocity as monitored by the velocity alarm on the hood. Gloves and safety glasses must be worn to avoid exposure through the skin and eyes from all reagents used. A lab coat is also recommended. Cracked, chipped or broken glassware must not be used.

Sample Collection, Preservation and Handling:

Sample Type	Preservation	Container	Holding time, collection to extraction	Holding time, extraction to analysis
Soil/Sediment	None; 4° C $\pm$ 2° C	500 mL glass bottle	14 days	40 days
Waste, solid	None; 4° C $\pm$ 2° C	500 mL glass bottle	14 days	40 days

8. Procedure:

Organization and Set-up:

8.1 Remove the samples to be extracted from the refrigerator. This set of samples and

corresponding QC samples are the extraction batch. Each extraction batch can include up to twenty samples, and must include a Laboratory Method Blank (LMB), laboratory fortified blank (LFB), matrix spike (MS) and matrix spike duplicate (MSD) of a sample.

8.2 Sample weight extracted will be approximately 10.0 g. The LMB and LFB will consist of approximately 10.0 g of Ottawa Sand. They are treated in the same manner as the samples.

8.3 Remove the appropriate surrogate(s) and spike(s) from the freezer as determined from the Surrogate and Spike Chart.

8.4 For each sample to be extracted, rinse a clean dry, 250 mL Pyrex beaker three times with methylene chloride. Allow the beakers to air dry in the hood.

Sample Preparation:

8.5 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. See KAR SOP KQ462 for specific procedure for percent solids determination.

8.6 Grind or otherwise reduce the particle size of the waste so that it is free flowing. Sufficient mixing or grinding can be accomplished using ether a wooden tongue depressor or a metal spatula. If a metal spatula is used it must be decontaminated between samples by washing with soap and water, followed by acetone and methylene chloride rinses.

8.7 Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction.

8.8 Place a rinsed beaker on the balance and TARE (this will reset the scale to 0.0 g, accounting for the weight of the beaker).

8.9 Weigh approximately 10.0 $\pm$ 0.1 g of prepared sample into one of the prepared beakers using a wooden tongue depressor. Record the exact weight of sample used on the Extraction Batch Data Sheet.

8.9.1 Tare beaker with sample. Weigh approximately 5.0 $\pm$ 0.5 g Celite® into the beaker. Record the exact weight on the Extraction Sheet. Slightly more (+1.0 g), or less (-1.0 g) of Celite® may be used depending on how much moisture is in the sample. The purpose is to produce a free flowing sample that is as dry as possible. Stir the sample and Celite® together (using the same wooden tongue depressor used to weigh the sample) until both are free flowing and well mixed.

8.10 Place a 1.91 cm glass fiber filter in the bottom a clean, methylene chloride rinsed cell. Transfer the sample from the beaker to the cell with the aid of a pyrex glass

powder funnel. If only one funnel is used, it must be washed and solvent rinsed between samples according to the same procedure used on the metal spatula.

8.11 Using the appropriate volume gas tight syringe add surrogate(s) to sample. Refer to Surrogate and Spike Chart for exact volume to add.

8.12 If the sample is a MS, MSD, or LFB, add the appropriate spike(s) in the same manner as the surrogate. Refer to Surrogate and Spike Chart.

8.13 Immediately cap the cell and place it in position on the ASE unit. Take a 60 mL glass vial with PTFE lined screw cap and label it with the sample ID using a Sharpe® marker. Place the labeled vial in the corresponding position to the cell on the ASE unit.

8.14 Repeat the process for each sample, MS and MSD.

8.15 Repeat the process for the LMB and LFB substituting 10 ± 0.5 g of Ottawa Sand for the sample.

8.16 ASE Extraction Conditions (stored under Methods):

Method 1 - All semivolatiles

Solvent:	Methylene Chloride
Oven temperature:	95° C
Pressure:	1800 psi
Static time:	6 min (after 5 min preheat equilibration)
Flush volume:	60% of the cell volume
Nitrogen purge:	180 seconds @ 150 psi

Extract Samples:

8.17 Turn on the nitrogen gas for the ASE unit. Regulator pressure should be 150 - 180 psi.

8.18 Make sure the 2 L solvent bottle is at least ½ full. If not, refill with methylene chloride.

8.19 Check the purge vial for waste, emptying it if it is ½ full or more.

8.20 Empty the three rinse vials of waste solvent and replace the PTFE lined septa.

8.21 Turn on the power to the ASE unit. Load Method 1. Press the **RINSE BUTTON,** this will perform an initial rinse of the system.

8.22 Load the appropriate starting vial number. Press **START**. Each sample extraction will take approximately 15 minutes. Allow the extracts to cool after completion. Place cooled extracts in the refrigerator until cleanup, concentration, or analysis is performed (See the appropriate KAR SOP).

9. Routine Preventative Maintenance:

9.1 Routine maintenance procedure for the ASE unit include:

9.1.1 Emptying and washing the cells and caps of spent soil/Celite®/ Hydromatrix® samples. Filling the solvent reservoir as needed, and emptying the waste vials as needed. Also, watch for leaks from the solvent reservoir, pumps, and valves. Any maintenance or repairs on the ASE unit itself must be noted in the ASE maintenance log book, along with the initials of the person performing the task and the date(s) performed.

9.1.2 Periodic maintenance on the ASE unit consists of:

9.1.2.1 Replacing the cell caps external o-rings (P/N 049457) 75 when worn.

9.1.2.2 Replace the PEEK seals (P/N 049455) inside all caps or when worn.

10. Quality Control:

10.1 Before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference - free. This is accomplished through the analysis of a solid matrix method blank (e.g., clean sand and clean Celite® or Hydromatrix®). Each time samples are extracted or when there is a change in reagents, a method blank needs to be extracted and analyzed for the compounds of interest. The method blank should be carried through all stages of the sample preparation and measurement.

Signature Page

To the best of my knowledge all information contained in this document is complete and accurate.

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QA Approval: Linda Felcyn Date: March 9, 2007
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STANDARD OPERATING PROCEDURE

Diesel Range Organics - Total Petroleum Hydrocarbons by GC/MS (Method 8270)

1.0 References:

1.1 Method 8270C: Test Methods for Evaluating Solid Wastes, Third Edition, Update III, SW-846; U.S. EPA Office of Solid Waste and Emergency Response, Washington, DC., December, 1996.

1.2 Wisconsin DNR Modified DRO Method: Method for Determining Diesel Range Organics; Wisconsin DNR, September, 1995.

1.3 "Turbovap Concentration," KAR Laboratories SOP KO3500B.

1.4 "Separatory Funnel Extraction by EPA Method 3510C," KAR Laboratories SOP KO3510.

1.5 "METHOD EPA 3535 Solid Phase Extraction (SPE)" KAR Laboratories SOP KO3535.

1.6 "Accelerated Solvent Extraction (3545)," KAR Laboratories SOP KO3545.

1.7 "Waste Dilution by EPA Method 3580A" KAR Laboratories SOP KO3580A.

1.8 "The Determination of Dry Weight Solids at 103-105° C," KAR Laboratories SOP KQ463.

1.9 "The Gravimetric Determination of Moisture and Total Solids at 103 - 105° C," KAR Laboratories SOP KQ462.

2.0 Application:

2.1 This method can be used to determine the concentration of Diesel Range Organics (DRO) in groundwater, TCLP leachates, soils, sediments, waste, and solid sample extracts. DRO is defined as all the chromatographic response (based on the quantitation ion m/z 57) falling between the onset of the n-decane (n-C_{10}) peak and the conclusion of the n-octacosane (n-C_{28}) peak. The attached chromatogram shows the m/z 57 ion current profile for DRO, and the total ion chromatogram of the internal standards, surrogates, and diesel component standard. Accuracy, precision, and method detection limit (MDL) data for DRO are listed in Table I.

3.0 Summary of Method:

3.1 A measured volume or weight of sample is extracted using an appropriate extraction procedure. The extract is dried, concentrated to a volume of 1.0 mL and injected into a GC/MS system. The compounds of interest are separated with a narrow-bore fused-silica capillary column during a temperature-programmed sequence, and are then detected with a mass spectrometer. Qualitative identification of the target compounds in the extract is based on the retention time and the mass spectra determined from standards analyzed on the same GC/MS system under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3.2 Sample collection, preservation and handling:

Matrix	Preservation/ Storage	Container	Sample Holding Time	Extract Hold Time
Aqueous	none; 4°C+/-2°C	1-L amber	7 days	40 days
Soil/sediment	none; 4°C+/-2°C	500mL	14 days	40 days
Waste	none; 4°C+/-2°C	Glass	14 days	40 days
TCLP	none; 4°C+/-2°C	1-L amber	7 days from TCLP leaching procedure	40 days

3.3 Store the sample extracts at -10° C, protected from light, in sealed vials (e.g. screw-cap vials or crimp-capped vials) equipped with unpierced PTFE-lined caps.

4.0 Safety:

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest level possible. Lab coats, gloves and lab glasses or face shield should be worn while handling extracts and standards. Standard preparation, addition of the internal standard solution, and sample extract dilution should be performed in a hood or well ventilated areas.

4.2 Material Safety Data Sheets (MSDS) are available to the analyst in each lab division. These sheets specify the type of hazard that each chemical poses and the procedures that are used to handle these materials safely.

5.0 Interferences:

5.1 Method interferences may be caused by contaminants in solvents, reagents or glassware. Glassware must be properly cleaned with hot soapy water and rinsed with both deionized water and extraction solvent. Any non-target interferences present at

sufficiently large amounts as to make DRO quantitation and identification impossible at the reported limit may be addressed by repeating the extraction and/or analysis, attempting sample clean-up, or diluting the sample extract and/or elevating the quantitation limit.

5.2 Matrix interferences may be caused by contaminants that are extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interference. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. If a sample is analyzed containing high concentrations of DRO (>1000 ppm), the sample must be followed by analysis of a solvent blank to check for carry-over before additional samples may be analyzed.

6.0 Apparatus & Materials:

6.1 Gas chromatograph - Thermofinnigan Trace GC with autosampler, splitless injector with digital pressure and flow control
Autosampler - Thermofinnigan Triplus: Injection Volume = 1µL
Mass spectrometer - Thermofinnigan Trace DSQ Quadrapole GC-MS
Capillary column - Phenomenex ZB-5 Guardian, 30 m x 0.25 mm ID, 0.25µm film thickness with built in 5 m guard column
Data system - Thermofinnigan Xcalibar and Chemstation Environquant software
Syringes - 1 mL, 2 mL, 25 µL 10 µL, 100 µL
10 mL vials with PTFE-lined screw caps
Autosampler vials and crimper, 1.5 mL with Teflon crimp caps
Volumetric flasks, class A, 10 mL with glass stoppers

7.0 Reagents & Standards:

Acetone - pesticide residue grade
Methylene chloride - pesticide residue grade

7.1 Stock standards/surrogates and spike solutions - purchased from Restek or Supelco as certified solutions; replace annually. Replace sooner if comparison with quality control check samples indicates a problem.

7.2 Calibration Stock Standards:
Diesel Fuel #2 composite Standard (31053) @ 5000µg/mL in methylene chloride

7.3 Tuning/Internal/Surrogate Stock Standards:
- Semivolatiles Base/Neutrals Surrogate Spike Mix @ 5000 µg/mL in methylene chloride (4-7262)
- Semivolatiles Internal Standards Stock Mix @ 2000 µg/mL in methylene chloride (4-8902)
- Semivolatiles GC/MS Tuning Stock Standard @ 1000 µg/mL in methylene chloride (GCM-150)

7.4 Matrix Spike Stock Standards:
Fuel Oil #2 @ 20,000 µg/mL in methanol (Supelco, 47594-U)

7.4.1 Transfer the stock standard solutions into vials with PTFE-lined screw-caps. Store, protected from light at -10° C or less. Stock standard solutions should be replaced annually, or sooner if signs of degradation appear. Check stock solutions frequently for signs of evaporation or degradation and replace as necessary.

7.5 Calibration Standards:

7.5.1 Five calibration standards are prepared for the initial calibration. The concentrations of these standards are 100 µg/mL, 200 µg/mL, 300 µg/mL, 400 µg/mL and 500 µg/mL, which define the working range of the detector. Each standard is prepared in methylene chloride so that the final volume of each is 1.0 mL.

7.5.2 The following equation is used to determine the volume of a stock standard needed to prepare a given volume of a calibration standard:

$$Vc = \frac{Ccal \times Vcal}{Cs}$$

where

Vs = volume of stock standard required to prepare the calibration standard (mL)
Cs = concentration of the stock standard (µg/mL)
Ccal = concentration of the calibration standard to prepare (µg/mL)
Vcal = volume of calibration standard to prepare (mL)

7.5.3 EXAMPLE:

Prepare 1 mL of a 300 µg/mL standard from the 5000 µg/mL stock standard

$$Vs = \frac{300\mu g/mL \times 1mL}{5000\mu g/mL} \doteq 0.060mL = 60\mu L$$

Calibration Standards Prep Table: amount of each to add to methylene chloride in 1 mL vial

Stock Concentration (µg/mL)	Calibration Standard Concentration (µg/mL) 100	200	300	400	500
Diesel #2 std.@ 5000	20 µL	40 µL	60 µL	80 µL	100 µL
Internal Std.@ 2000	20 µL	20 µL	20 µL	20 µL	20 µL
B/N Surr.@ 5000	20 µL	40 µL	60 µL	80 µL	100 µL
Methylene Chloride	940 µL	900 µL	860 µL	820 µL	780 µL

7.5.4 Calibration standards may be used for up to 12 months stored at -5 to -20° C.

7.5.5 GC/MS tuning standard - A methylene chloride solution containing 50 ng/µL of decafluorotriphenyphosphine (DFTPP) should be prepared. This is prepared by drawing-up 950 µL of methylene chloride using a 1.0 mL syringe and placing it in a 1.0 mL crimp seal PFTE-lined vial. Using a 100 µL syringe, draw-up 50 µL of the semi-volatiles GC/MS tuning stock standard and add it to the 950 µL of methylene chloride in the vial. The tuning standard may be used for up to 6 months stored at -5 to -20° C.

8.0 Sample Preparation:

8.1 The sample extraction procedures are given in the following SOPs:

Matrix	SOP Number	Extraction Technique
Aqueous	KO3535	Solid Phase Extraction
Aqueous, TCLP leachates	KO3510	Separatory Funnel
Soils/Sediments	KO3545	Accelerated Solvent Extraction
Wastes	KO3580	Waste Dilution

8.2 The sample concentration procedures are given in KO3500B.

9.0 Procedure:

9.1 Instrument Conditions:

Column: Phenomenex ZB-5 Guardian, 30 m x 0.25 mm ID, 0.25µm film thickness with built in 5 m guard column
Column flow: Approximately 2.5 mL/min helium
GC Oven temperatures:
Initial column temperature: 40° C for 4 minutes
Column temperature program: 10° C per minute
Final column temperature: 320° C
GC injector parameters:
Injector temperature: 240° C
Injector: splitless
Injector purge time: 0.8 minutes
Injector liner: 4mm ID quartz or 4mm glass, deactivated
Sample injection volume: 1 µL
Mass Spectrometer and interface parameters:
Mass spectrometer interface: 270° C
Manifold temperature: 200° C
Mass range: 45-550 amu
Scan rate: 1600 amu/sec
Emission current: 100 micro amps
A/M amplitude Voltage: 3.5 volts

9.2 Tune and Calibration Procedure:

9.2.1 Fifty nanograms of DFTPP must be analyzed at the beginning of each 12 hour clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria demonstrates that the instrument is measuring the proper masses in the proper ratios. The DFTPP analysis takes place under the same instrument conditions as the calibration standards and samples except that a starting GC temperature of 160° C is used to allow for a faster elution of DFTPP. All other instrument conditions must be identical; the mass range, scan rate, and multiplier voltage. Analyze a 1 µL aliquot of the 50ng/µL DFTPP solution. Evaluate the DFTPP peak. The chromatogram should exhibit acceptable baseline behavior and the DFTPP peak should be symmetrical.

9.2.2 The following criteria for acquiring the mass spectrum of DFTPP are required: average the spectrum across the peak and background subtract by using a single scan acquired no more than 50 scans prior to the eluting DFTPP peak. The background subtraction should be designed to eliminate column bleed or background ions only. The abundance criteria listed below must be met before standard analysis and subsequent sample analysis can proceed.

Mass (m/z)	Abundance Criteria
51	10-80% of the base peak
68	<2% of mass 69
69	Exists only
70	<2% of mass 69
127	10-80% of the base peak
197	<2% OF MASS 198
198	Base peak or >50% of mass 442
199	5-9% of mass 198
275	10-60% of the base peak
365	>1% of the base peak
441	Present and less than mass 443
442	Base peak or >50% of mass 198
443	15-24% of mass 442

NOTE: The criteria above have been taken from Method 525.2, Determination of Organic Compounds in Drinking Water By Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry, and represent modern performance criteria that reflect the improved mass spectrometers in use today.

9.2.3 The following records must be kept for each DFTPP analysis that meets the criteria:

-the date, time, and data file of the analysis
-a spectrum of the scan or enhanced scans
-a tabulation of the ion abundance of the scan or enhanced scans

9.2.4 If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA).

9.2.5 Target tune parameters for PFTBA are:

m/z 69:	100%
m/z 70:	1.5-3%
m/z 131:	20-50%
m/z 132:	1.5-3%
m/z 219:	20-60%
m/z 220;	1.5-3%
m/z 414:	present
m/z 464:	present
m/z 502:	> 0.8%

9.2.6 Depending on the nature of the results for the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

9.2.7 After the DFTPP criteria have been met, the initial calibration standards are analyzed.

9.2.8 The initial calibration consists of five points, encompassing the linear range of the instrument. The concentrations are: 100 µg/mL, 200 µg/mL, 300 µg/mL, 400 µg/mL, and 500 µg/mL. Set up a sequence and analyze the calibration standards. The injection volume must be the same for the calibration standards and all sample extracts, i.e. 1µL. Identify the internal standards, surrogates, and the DRO compounds. The DRO is quantitated by measurement of the total area, baseline-to-baseline, of all the m/z 57 ions over the range of C-10 thru C-28 peaks (approximately Scan 1000 - 3100). The data system must be updated with the proper retention times and ion data. The retention time window is defined as ± 0.06 RRT units of any of the base alkane peaks in the standard from their associated internal standard reference (see Table VI). The relative response factor for each surrogate and total DRO is calculated using the data system according to the following formula:

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where:

Ax = area of the characteristic ion for the compound being measured

Ais= area of the characteristic ion for the internal standard associated with the compound being measured. (See Table VI for a list of the compounds that are associated with each internal standard.)

Cx = concentration of the compound being measured (µg/mL)

Cis = concentration of the internal standard (40 µg/mL)

9.2.9 The average relative response factor (RRF_{avg}) is calculated for DRO and each surrogate compound:

$$RRF_{avg} = \frac{RRF1 + RRF2 + RRF3 + \ldots + RRFn}{n}$$

RRF1 = relative response factor of the first standard
RRFn = relative response factor of the last standard
n = number of calibration standards

9.2.10 Calculate the standard deviation (SD) for the initial calibration standards:

$$SD = \sqrt{\sum_{i-1}^{n-1} \frac{(RRF_1 - RRF_{avg})^2}{n-1}}$$

9.2.11 Calculate the relative standard deviation (%RSD) of DRO in the calibration standards:

$$\%RSD = \frac{SD}{RRF_{avg}} \times 100$$

9.2.12 The results are evaluated for linearity according to the following criteria: If the RSD is less than or equal to 20%, the calibration is assumed linear through the origin and the average response factor can be used for quantitation.

9.2.13 If the initial calibration criteria are not met (%RSD>20), action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port liner and assembly. In any case, if the criteria are not met, the initial calibration must be repeated. The analyst must be aware of the 12-hour clock for the DFTPP analysis. The DFTPP criteria must be met prior to the analysis of the calibration standards.

9.2.14 Samples are analyzed only after the DFTPP criteria and the calibration acceptance criteria have been met. The analytical system must be evaluated every 12 hours by the analysis and evaluation of the DFTPP and a mid-level calibration standard (the 300 µg/mL standard).

9.3 Analysis Sequence

INITIAL CALIBRATION	CONTINUING CALIBRATION
DFTPP 50ng on column Clock starts at injection	DFTPP 50ng on column Clock starts at injection
Calibration standards	Mid point calibration verification
Samples analyzed until the 12-hour clock expires	Samples analyzed until the 12-hour clock expires

9.3.1 The percent difference or percent drift between the continuing calibration RRF and the average relative response factor (RRF_{avg}) is calculated for DRO and each surrogate compound:

$$\%difference = \left| \frac{RRF - RRF_{avg}}{RRF_{avg}} \right| \times 100$$

where:
RRF = relative response factor from CCV
RRF_{avg} = average relative response factor from initial calibration curve

$$\%drift = \left| \frac{Ccurve - Ct}{Ct} \right| \times 100$$

where:
Ccurve = concentration from curve (µg/mL)
Ct = true concentration of CCV (µg/mL)

9.3.2 The continuing calibration is evaluated for DRO. The %D (Drift) criterion for DRO must be met before the analysis of sample extracts can begin.

Continuing Calibration: DRO: <20% difference from initial calibration

9.3.3 If the continuing calibration criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of the injection port lines and assembly. In any case, if the criteria are not met, the analysis of the continuing calibration standard must be repeated. The analyst must be aware of the 12-hour clock for the DFTPP analysis. The DFTPP criteria must be met prior to the analysis of the calibration standards.

10.0 Sample Analysis/Calculations:

10.1 Remove the sample extracts from the refrigerator and allow them to come to ambient temperature prior to analysis.

10.2 Set up a sequence table for the samples and analyze them under the same conditions as the tune and standards.

10.3 If a sample is analyzed which contains DRO concentrations above 1000 µg/mL, the sample must be diluted and reanalyzed.

10.4 The dilution factor is calculated by dividing the volume of sample extract in microliters into 1000. For example, if 100 µL of a sample extract is diluted to final volume of 1.0 mL, the dilution factor is 10. (1000/100=10). The following table gives some dilution factors:

Dilution Preparation

µL extract-Vext	µL $MeCl_2$	volume of dilution (Vdil-µL)	µL ISTD (2000µg/mL)-Vist	DF
1000	0	1000	20	1
500	500	1000	10	2
200	800	1000	16	5
100	900	1000	18	10
50	950	1000	19	20
20	980	1000	20	50

10.5 The concentration of internal standards must remain constant for all extracts and extract dilutions at 40 µg/mL. The following equation can be used to determine the volume of the 2000µg/mL internal standard solution to add to an extract when a dilution is prepared from an extract that has already been spiked with the internal standard solution:

$$V_{istd}(\mu L) = 20\mu L - \left(\frac{V_{ext}}{V_{dil}} \times 20\mu L\right)$$

where: Vistd = volume of 2000µg/mL internal standard to add to the diluted extract (µL)
Vext = volume of extract used to prepare the dilution (µL)
Vdil = final volume of the dilution (µL) (1000 µL or 1.0 mL)

10.6 Qualitative Analysis for DRO :

10.6.1 DRO is identified by the visual comparison of the sample mass spectrum with the mass spectrum of DRO from the daily calibration standard, and by their respective retention times in the chromatogram. It is important to note that no specific analyte is being identified, but rather all compounds with a base hydrocarbon molecule eluting within the DRO range of the chromatograph. Although no specific analytes are identified by spectra, several of the n-Alkanes should be directly referenceable to the standard, and are present in most forms of DRO found in samples.

10.6.2 Quantitative Analysis for DRO:

Calculations for Samples-Internal Standard Technique
These calculations assume that the same volume is injected for standards and samples.

Aqueous samples - the calculation for samples is:

$$concentration(\mu g / L) = \frac{Ax}{Ais} \times \frac{Cis}{RRF_{avg}} \times \frac{F}{V} \times DF$$

where:
Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (µg/L)
RRF_{avg} = average response factor of the compound being measured
F = final volume of extract (mL)
V = volume of sample extracted (L)
DF = dilution factor

10.7 The reporting limit (RL) for each sample is given:

$$RF(\mu g / L) = 10 \times \frac{F}{F_{qap}} \times \frac{V_{qap}}{V} \times DF$$

where:
F = final volume of extract (mL)
Fqap = 1.0 mL
Vqap = 1.0 L
V = volume of sample extracted (L)
DF = dilution factor.

NOTE: If V=800 mL to 1200 mL, assume that Vqap/V = 1 in the calculation of the reporting limit.

10.8 Soils - the calculation for samples is:

$$concentration(\mu g / kg, dw) = \frac{Ax}{Ais} \times \frac{Cis}{RRF_{avg}} \times \frac{F}{(W)(solids)} \times DF$$

where:

Ax = area of the characteristic ion of the compound being measured
Ais = area of the characteristic ion of the internal standard
Cis = concentration of the internal standard (µg/mL)
RRF_{avg} = average response factor of the compound being measured
F = final volume of extract (mL)
W = weight of sample extracted (kg)
solids = (percent solids)/100
DF = dilution factor

10.9 Refer to KAR SOP KQ463 or KQ462 for details on determining percent solids.

10.10 The reporting limit (RL) for each sample is given:

$$RL = 300 \times \frac{F}{F_{qap}} \times \frac{W_{qap}}{(W)(solids)} \times DF$$

where:

F = final volume of extract (mL)
W = weight of sample extracted (kg)
solids = (percent solids)/100
DF = dilution factor
F_{qap} = 1.0 mL
W_{qap} = 10.0 g

10.11 The concentrations of target compounds in the lab fortified blank and matrix spike samples are determined in the same manner as that of the samples, explained in the preceding paragraphs. These concentrations are then compared to the theoretical spike concentrations and the percent recovery is calculated. The true value for a spiked sample is calculated as follows:

$$concentration = \frac{amounts\ of\ compound\ added}{Volume/weight\ of\ sample\ extracted}$$

10.12 For liquid samples, this equation expands to:

$$Concentration(\mu g / L) = \frac{Cspike \times Vspike}{Vsample}$$

where:

Cspike = concentration of the spiking solution (µg/mL)
Vspike = volume of spiking solution added to the sample (mL)
Vsample = volume of sample spiked (L)

10.13 For soil and solid samples, the equation expands to:

$$Concentration(\mu g / kg) = \frac{Cspike \times Vspike}{Wsample \times solids}$$

where:

Cspike = concentration of the spiking solution (µg/mL)
Vspike = volume of spiking solution added to the sample (mL)
Wsample = weight of sample spiked (kg)
solids = (percent solids)/100

10.14 The percent recovery of the target analytes in the matrix spike sample and lab control samples is calculated:

$$\%recovery(\%REC) = \frac{(Cms - Cs)}{Ts} \times 100$$

where:

Cms = concentration of the spiked sample (MS/MSD or LCS/LCSD)
Cs = concentration of the unspiked sample
Ts = theoretical concentration of the spike

10.15 The matrix spike sample is analyzed in duplicate (MS/MSD). The relative percent different (%RPD), a measure of precision, is calculated for the MS/MSD pair as follows:

$$\%RPD = \frac{\%REC(MS) - \%REC(MSD)}{\left(\%REC(MS) + \%REC(MSD)\right)/2} \times 100$$

10.16 The absolute value of the %RPD is evaluated and reported; that is, only positive values are reported. If the MS/MSD cannot be performed, LCS/LCSD are analyzed and evaluated.

11.0 QA/QC:

11.1 The analytical batch consists of up to twenty client samples, prepared by matrix, and a method blank (LMB), matrix spike (MS), matrix spike duplicate (MSD) and a laboratory fortified blank (LFB). A laboratory fortified blank and laboratory fortified blank duplicate may be substituted if insufficient sample is available for MS/MSD.

11.2 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 extracted 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.

11.3 Initial Demonstration of Capability (IDOC):

11.3.1 Each analyst must demonstrate competence in the analysis of samples by this procedure. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made.

11.3.2 The criteria for this demonstration are the preparation and analysis of four replicate 1.0 L aliquots of spiked reagent water, containing DRO at approximate 2-10 times its MDL. By analyzing seven replicates, establishment of the MDL for DRO can be established concurrently with the IDOC.

11.3.4 To evaluate the performance of the total analytical process, the 7 spiked samples must be handled in exactly the same manner as actual samples. Use a clean matrix for spiking purposes (one that does not have any target or interference compounds), e.g. organic-free reagent water for the aqueous matrix and organic-free sand or soil for solid matrix.

11.3.5 The IDOC (or MDL) spike solution itself should contain DRO at 1000 µg/mL. Prepare this solution using the same formula for preparing the calibration standard, except prepare it in 5.0 mL methanol. To each of the seven 1.0 L aliquots of reagent water add exactly 1.0 mL of the IDOC/MDL spike solution using a 1.0 mL gas tight syringe. Process and analyze the IDOC /MDL samples according to the same procedures used to analyze and prepare actual samples.

11.3.6 Calculate the average recovery ($\bar{X}$), the standard deviation of the recovery(s), and the MDL for DRO (see Table I.).

11.3.7 Additional MDLs are performed whenever significant changes are made to instrumentation or personnel.

11.3.8 The MS, MSD, and LFB spike analyte concentrations and recovery data are listed in Table II. Recovery windows and % difference limits are generated by the laboratory through analysis over time according the SW-846, Method 8000, sec. 8.0. Surrogate compounds and recovery windows are listed in Table III. Acceptance criteria and corrective actions for all QC items are discussed in Tables IV and V.

12.0 Preventative Maintenance:

12.1 Routine maintenance of the GC/MS system is critical in order to produce quality data. Assessment of the system and possible maintenance operations to be performed is done daily when in use, based on evaluation of the previous day's samples and the current DFTPP and calibration analyses. All maintenance operations are logged in an Instrument Maintenance Logbook sequentially, per instrument. Typical daily maintenance operations include: changing the injector septum and liner; clipping off 12 inches of the column at the injector end to improve chromatography. Typical routine maintenance operations include: replacing the filament and cleaning the ion source. Scheduled maintenance includes: changing the turbo pump oil every six months; changing the high vacuum pump oil every three months.

Table I: DRO MDL Data by EPA 3545 (KAR SOP3545)

Replicate	True Value mg/kg	Measured Value mg/kg	Grubb's "T" Test (outlier if > 1.938)
1	10.0	9.091	1.362
2	10.0	10.957	1.313
3	10.0	9.674	0.527
4	10.0	10.762	1.033
5	10.0	9.371	0.961
6	10.0	10.137	0.137
7	10.0	10.297	0.367

Mean:	10.0143	**Ave. % Recovery:**	100.41
Std. Dev.	0.6976	**Rel. % Diff.:**	6.95
Signal/Noise:	14.39	**MDL:**	2.192505

Table II: MS, MSD, and LFB Concentrations and Recovery Windows

Spiking Mix	Sample Conc.		QC Limits, % Recovery		Max. RPD	
	Soil(mg/kg)	Aqueous(mg/L)	Soil	Aqueous	Soil	Aqueous
Fuel Oil #2 20,000µg/mL	50	0.50	60-140	60-140	30	30

Table III: Surrogate Compounds and Recovery Windows for DRO Analysis

Mix	Surrogate	QC Limits, % Recovery	
		Soil	Aqueous
B/N	Nitrobenzene-d5	23-120	35-114
B/N	2-Fluorobiphenyl	30-115	43-116
B/N	Terphenyl-d14	18-137	33-141

Table IV: QC Items and Corrective Actions

QC Item	Frequency	Acceptance Criteria	Corrective
Tune DFTPP 50ng	Prior to analysis of calibration standards every 12 hours	DFTPP - within criteria	-Reanalyze and evaluate -Retune and analyze -Clean source, retune, reanalyze
Initial Calibration	After Tune Check and when calibration verification standard fails acceptance criteria. All initial calibration standards.	%RSD <20%	-reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standard -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standard
Continuing Calibration Verification	After tune check; every 12 hours prior to analysis of samples	%Difference <= 20% or %Drift <=20%	-Reanalyze standard -prepare new standard and reanalyze -Recalibrate
Internal Standard Areas	Evaluate all standards and samples	Areas in continuing calibration verification must be 50% to +200% of previous midlevel standard Areas in samples should be evaluated for gross error . Consult superior	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available

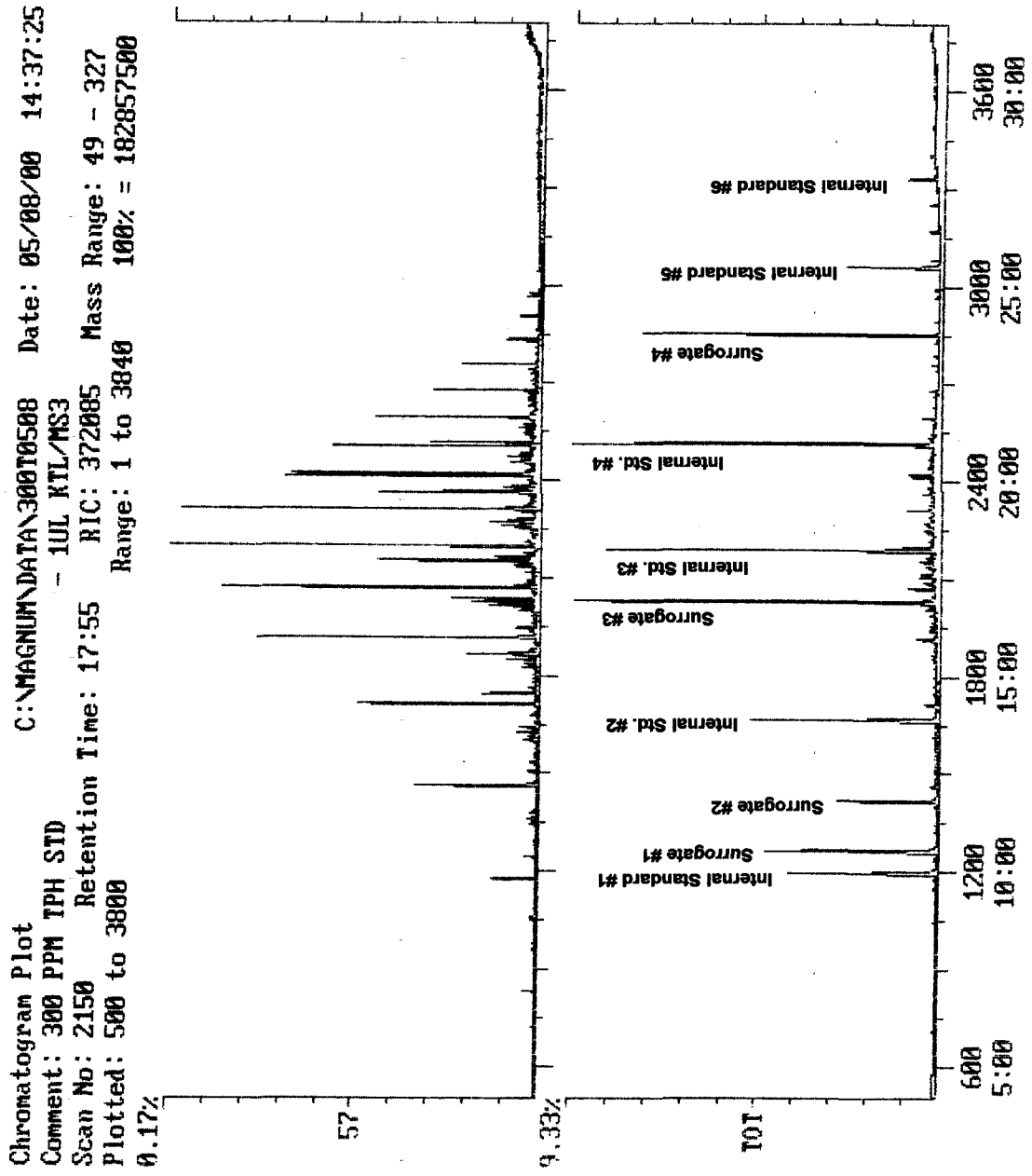
Table V (cont): QC Items and Corrective Actions

QC Item	Frequency	Acceptance Criteria	Corrective Action
Surrogate recovery	Evaluate for all samples and QC items if extract is not diluted OR If diluted, where >reporting limit	Within Table III limits; one may be outside of criteria	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract(s) -Re-extract and reanalyze if sufficient sample available
Method Blank	Per batch	All targets : <4.0 mg/kg soils; <0.1 µg/L waters	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Re-extract entire batch or explain in narrative
Lab Fortified Blank(LFB).	Per Batch	Within the accuracy limits established according to the procedure stated in the QA/QC section	-Evaluate chromatogram, spectra, and integrations -reanalyze extract
Matrix spike (MS) Matrix Spike Duplicate (MSD)	Per batch if sufficient sample volume/weight supplied	Within the control limits established according to the procedure stated in the QA/QC section	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract
Reporting Limit (RL)	Daily (optional)-lowest point on calibration curve if required by client.	Detected at reasonable sensitivity	-Evaluate integrations and spectra; reanalyze -Prepare new standard and reanalyze
Initial Demonstration of Capability (IDOC)	Per analyst	Accuracy and precision within method specified criteria	-Evaluate data -Reanalyze extracts if warranted -Re-extract and reanalyze for targets that fail criteria

Table VI: Retention Time Data, Quantitation Ions, and Internal Standard References for Method Analytes:

Compound	Retention Time (min:sec)	Relative Retention Time	Quantitation Ion	IS Reference #
Internal Standards				
1,4-dichlorobenzene-d4 (#1)	9:58	1.00	150	
naphthalene-d8 (#2)	13:53	1.00	136	
acenaphthene-d10 (#3)	18:15	1.00	164	
phenanthrene-d10 (#4)	20:58	1.00	188	
chrysene-d12 (#5)	25:32	1.00	240	
perylene-d12 (#6)	27:46	1.00	264	
Surrogates				
nitrobenzene-d5	11:46	0.85	82	2
2-fluorobiphenyl	16:55	0.93	172	3
terphenyl-d14	23:47	0.93	244	5
Analytes				
TPH C_{10}-C_{28}	9:00-28:00		57	4
decane C10	9:47	0.98	57	1
tetradecane C14	17:15	0.94	57	3
octadecane C18	20:54	1.00	57	4
eicosane C20	22:22	1.07	57	4
hexacosane C24	24:49	0.97	57	5

Attachment I: DRO chromatogram with Surrogates and Internal Standards



Signature Page

To the best of my knowledge all information contained in this document is complete and accurate.

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QA Approval: _____ Date: 19-Dec-2005
Pollyann Nee

STANDARD OPERATING PROCEDURE

Determination of Gasoline Range Organics by GC/MS by 8260B using HP GC-MS

1.0 References

1.1 U.S. EPA Method 8260B, SW-846, 3rd Edition, Update III, December, 1996.

1.2 Wisconsin DNR Modified GRO Method, PUBL.-SW-140, September, 1995.

1.3 "Closed-System Purge & Trap and Extraction for High-Level Volatile Organics," KAR Laboratories, Inc. SOP KO5035H.

2.0 Application

2.1 Water and soil samples with an undiluted concentration range of 100 - 1000 µg/L for water and 5 - 50 mg/kg for soils.

3.0 Summary of Method

3.1 This method employs the technique of purge and trap, coupled with a gas chromatograph/mass spectrometer. An aliquot of sample is purged in a gas tight chamber with UHP grade helium to extract the volatile compounds. The vapor is swept onto a sorbent trap where the volatiles are adsorbed. Next, the sorbent trap is heated and back flushed, thereby desorbing the volatiles onto the column within the gas chromatograph. The fused silica capillary column is then temperature programmed to separate the volatiles prior to detection by the mass spectrometer.

4.0 Interferences

4.1 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Carryover can be reduced by rinsing the sample syringe between loadings. When an unusually concentrated sample is encountered, it should be followed by the analysis of reagent water as a check of contamination.

4.2 Samples can be contaminated by the diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Use a trip blank consisting of reagent water carried through sampling and storage as a check for such contamination.

5.0 Apparatus & Equipment

5.1 System 1

- Hewlett Packard Model 5890 Plus gas chromatograph with HP-624 column, 25 m long x 0.20 mm ID
- Hewlett Packard Model 5972 mass selective detector.
- OI 4552 automatic sampler.
- Tekmar LSC-3000 sample concentrator with 10 cm Carbopack B, 6 cm Carboxen 1000, 1 cm Carboxen 1001 trap, Supelco 2-4920.
- Hewlett Packard Kayak XA with MS Chemstation and Enviroquant software package and NBS75K library.
- Hewlett Packard LaserJet Plus printer.

5.2 System 2

- Hewlett Packard Model 5890 Series II chromatograph with DB-624 column, 30 m long x 0.25 mm ID
- Hewlett Packard Model 5971 mass selective detector.
- OI 4552 automatic sampler.
- Tekmar LSC-3100 sample concentrator with 10 cm Carbopack B, 6 cm Carboxen 1000, 1 cm Carboxen 1001 trap, Supelco 2-1066.
- Hewlett Packard Kayak XA with MS Chemstation and Enviroquant software package and NBS75K library.
- Hewlett Packard LaserJet Plus printer.

5.3 System 3

- Hewlett Packard Model 5890 Plus gas chromatograph with DB-624 column, 25 m long x 0.20 mm ID
- Hewlett Packard Model 5972 mass selective detector.
- OI 4552 automatic sampler.
- Tekmar LSC-2000 sample concentrator with 10 cm Carbopack B, 6 cm Carboxen 1000, 1 cm Carboxen 1001 trap, Supelco 2-1066.
- ASUS Pentium III PC with 20 gig harddrive.
- Hewlett Packard MS Chemstation and Enviroquant software package.
- CSS Analytical NIST 98 Library.
- Hewlett Packard LaserJet 4100 printer.

5.4 Gas tight Syringes, 5 mL Luerlock with shut-off valve, Hamilton 1005.

5.5 Micro syringes:

10 µL, Hamilton 1701
25 µL, Hamilton 1702
100 µL, Hamilton 1710
250 µL, Hamilton 1725
500 µL, Hamilton 1750
1000 µL, Hamilton 1001

5.6 Volumetric flasks: 10, 50, 100, 500, 1000 mL with ground glass stoppers.

5.7 Analytical balance, OHAUS Model No. C305-S, 0.1mg sensitivity.

5.8 Glass scintillation vials, 7 mL and 20 mL with Teflon® -lined screw caps.

5.9 Pasteur pipets.

5.10 Mininert valves, Supelco 3-3300M.

6.0 Chemicals

6.1 Reagent Water, carbon filtered.

6.2 Methanol, Purge & Trap grade, Burdick & Jackson 232-1.

6.3 Gases:

Purge Gas: He, 99.998%

Carrier Gas: He, 99.998%

6.4 Assayed individual standard materials

Fluorobenzene

1,2-Dichlorobenzene-d4

1,2-Dichloroethane-d4

Toluene-d8

4-Bromofluorobenzene

6.5 Certified standard mixtures:

Unleaded Gasoline Composite Standard, Restek Corporation 30081 (2500 μg/mL).

7.0 Standard Preparation

7.1 Stock Standards : Prepare a stock standard solution for the individual standard materials as follows:

7.1.1 Place 80-90 mL methanol in a 100 mL tared volumetric flask. Allow the flask to stand unstoppered until all alcohol-wetted surfaces have dried. Stopper and weigh the flask to the nearest 0.1 mg.

7.1.2 For analytes which are liquids at room temperature, add approximately 500 μL of the reference standard to the methanol using a 1000 μL syringe. Make sure that the material falls directly into the alcohol without contacting the sides of the flask.

7.1.3 Quickly stopper the flask and reweigh.

7.1.4 Repeat sections 7.1.2 and 7.1.3 for each component to be included in the stock solution. The 1000 μL syringe should be rinsed with methanol and the next standard material before the next addition to the flask.

7.1.5 When all the stock standard components have been added and individual weights obtained, dilute to volume with methanol, stopper and mix by inverting several times.

7.1.6 Store the stock standard solution in a 100 mL volumetric flask. Solution is stable for 1 year when stored at -20°C.

7.1.7 Calculate the concentration of each component in μg/mL from the net gain in weight.

NOTE: THE WEIGHT OF COMPOUNDS WITH A CERTIFIED PURITY OF 96% OR GREATER CAN BE USED WITHOUT CORRECTION IN CALCULATING THE CONCENTRATION OF THE STOCK STANDARD. Each component concentration should be approximately 5000 μg/mL (will vary with component density and response).

7.2 Secondary Internal Standard / Surrogate standard from stock standard prepared in section 7.1:

7.2.1 Place 50 mL methanol into a 100 mL volumetric flask.

7.2.2 Pipette 5.0 mL or 0.5 mL of the stock solution into the 100 mL volumetric flask. The 5.0 mL addition is used to prepare a 250 µg/mL standard and the 0.5 mL addition is used for a 25 µg/mL standard.

7.2.3 Dilute to volume with methanol and invert each of the flasks several times to mix.

7.2.4 Solution is stable for 6 months when stored at room temperature.

	Approximate Component Concentration
IS/Surrogate Mixture	250 µg/mL or 25 µg/mL*

* Will vary with component weight as determined in Step 7.2.2

7.2.5 The final instrumental concentration of the 250 µg/mL standard will be 50 µg/L while the final instrumental concentration of the 25 µg/mL standard will be 5.0 µg/L.

8.0 Procedure

8.1 Instrument Conditions:

8.1.1 System 1

8.1.1.1 Environmental Data Analysis conditions:

Audit trail: on

8.1.1.2 GC operating conditions:

Carrier gas rate: He @ 0.6 mL/min
Initial temperature: 45°C for 4 minutes
Temperature program: 8° C/min to 200° C
Final temperature: 200° C for 5 minutes
Vacuum compensation: On
Pressure: 8.7 psi
Split ratio: 33:1
Injector temperature: 220° C

8.1.1.3 MS operating conditions:

Mass range: 45-260 amu
Threshold: 50
Scan rate: 1.9 scans/sec
Source temperature: 280° C

8.1.1.4 Purge & Trap conditions:

Purge temperature: 35° C
Purge flow: 40 mL/min
Sample heater: On
Prepurge time: 0 minutes
Sample preheat time: 1 minute
Sample preheat temperature: 40° C
Purge time: 11 minutes
Dry purge time: 1 minute
Desorb flow rate: 20 mL/min
Desorb preheat temperature: 245° C
Desorb time: 4 minutes
Desorb temperature: 250° C

Bake time: 15 minutes
Bake temperature: 260° C
Bake gas bypass: On
Bake gas bypass delay time: 2 minutes

8.1.1.5 Autosampler conditions:
Sample Type: Water
First Vial:
Last Vial:
Sample Volume: 5 mL
Dilution Factor: 0
Rinse Volume: 5 mL
Rinses: 2
Standard 1: Yes
Standard 2: No
S. Preheat Stir: No
Stir: No
W. Stir Time: 0 minutes
W. Settle Time: 0 minutes
Syringe Flushes: 2
PreHeat: No
Purge Time: 11 minutes
Desorb Time: 4 minutes
Cycle Timer: 40 minutes
Link to Method: 00

8.1.2 System 2

8.1.2.1 Environmental Data Analysis conditions:
Audit trail: on

8.1.2.3 GC operating conditions:
Carrier gas rate: He @ 0.94 mL/min
Initial temperature: 35° C for 3 minutes
Temperature program: 8°C/min to 200° C
Final temperature: 200° C for 3 minutes
Vacuum compensation: On
Split ratio: 35.2:1
Injector temperature: 220° C

8.1.2.3 MS operating conditions:
Mass range: 45-260 amu
Threshold: 50
Scan rate: 1.9 scans/sec
Source temperature: 280° C

8.1.2.4 Purge & Trap conditions:
Purge temperature: 35° C
Purge flow: 40 mL/min
Sample Heater: On
Prepurge time: 0 minutes
Sample Preheat temperature: 40° C
Purge time: 11 minutes

Dry purge time: 1 minute
Desorb flow rate: 20 mL/min
Desorb preheat temperature: 245° C
Desorb time: 4 minutes
Desorb temperature: 250° C
Bake time: 10 minutes
Bake temperature: 260° C

8.1.2.5 Autosampler conditions:

Sample Type: Water
First Vial:
Last Vial:
Sample Volume: 5 mL
Dilution Factor: 0
Rinse Volume: 5 mL
Rinses: 2
Standard 1: Yes
Standard 2: No
S. Preheat Stir: No
Stir: No
W. Stir Time: 0 minutes
W. Settle Time: 0 minutes
Syringe Flushes: 2
PreHeat: No
Purge Time: 11 minutes
Desorb Time: 4 minutes
Cycle Timer: 40 minutes
Link to Method: 00

8.1.3 System 3

8.1.3.1 Environmental Data Analysis conditions:

Audit trail: on

8.1.3.2 GC operating conditions:

Carrier gas rate: He @ 0.6 mL/min
Initial temperature: 35° C for 4 minutes
Temperature program: 8°C/min to 200° C
Final temperature: 200° C for 5 minutes
Vacuum compensation: On
Pressure: 9.0 psi
Split ratio: 33:1
Injector temperature: 220° C

8.1.3.3 MS operating conditions:

Mass range: 45-260 amu
Threshold: 50
Scan rate: 1.9 scans/sec
Source temperature: 280° C

8.1.3.4 Purge & Trap conditions:

Purge temperature: 45° C
Purge flow: 40 mL/min
Sample heater: On

Prepurge time: 0 minutes
Sample preheat time: 1 minute
Sample preheat temperature: 40° C
Purge time: 11 minutes
Dry purge time: 1 minute
Desorb flow rate: 20 mL/min
Desorb preheat temperature: 245°C
Desorb time: 4 minutes
Desorb temperature: 250° C
Bake time: 15 minutes
Bake temperature: 260° C
Bake gas bypass: On
Bake gas bypass delay time: 2 minutes

8.1.3.5 Autosampler conditions:

Sample Type: Water
First Vial:
Last Vial:
Sample Volume: 5 mL
Dilution Factor: 0
Rinse Volume: 5 mL
Rinses: 2
Standard 1: Yes
Standard 2: No
S. Preheat Stir: No
Stir: No
W. Stir Time: 0 minutes
W. Settle Time: 0 minutes
Syringe Flushes: 2
PreHeat: No
Purge Time: 11 minutes
Desorb Time: 4 minutes
Cycle Timer: 40 minutes
Link to Method: 00

8.2 Calibration standard preparation:

8.2.1 Prepare a series of 5 calibration standards by injecting the volumes of the secondary Unleaded Gasoline Composite calibration solution into 100 mL volumetric flasks filled to volume with reagent water.

Level	µL/100 mL	Concentration
125	5	125ppb
250	10	250ppb
500	20	500ppb
750	30	750ppb
1000	40	1000ppb

8.2.2 Mix by inverting the flasks gently three times.

8.3 QC Check Sample preparation:

8.3.1 Inject 10 µL of the secondary Unleaded Gasoline Composite calibration

solution into a 100 mL volumetric flask filled to volume with reagent water.

8.3.2 Mix by inverting the flasks gently three times. Approximate component concentration: 250 μg/L.

8.4 Soil Preparation (see SOP KO5035H for extraction procedure of soils).

8.4.1 Using a glass pipet withdraw 1.0 mL of methanol extracted sample and add to a 50 mL glass volumetric half filled with reagent grade water. Dilute to volume.

8.4.2 Add the contents of the 50 mL volumetric to a 40 mL VOA vial.

8.5 Water Preparation.

8.5.1 Load the first sample into position #1 on the autosampler.

8.5.2 Repeat for all samples.

8.5.3 Press Program Setup on the autosampler and step through the autosampler conditions using the Program Setup button. Confirm the autosampler conditions in Instrument Conditions of this section and enter the Last sample loaded on the autosampler tray.

8.5.4 Check the internal standard/ surrogate level and add more if necessary.

8.5.5 Press the Auto Mode button to initiate the analytical run.

8.6 Programming the run for samples:

8.6.1 Select WINDOWS file of ENVTop#1. Instrument 1 MS Top window should appear.

8.6.2 Select SEQUENCE, LOAD and file DEFAULT.s.

8.6.3 Select RUN. Change the Data File Directory to reflect the current date, with the entire directory D:/HPCHEM/1/DATA/MMDDYY.D., where MM, DD and YY represent the current month, day and year. Select OK.

8.6.4 Select EDIT SAMPLE LOG TABLE. The Sample Log Table window will appear. Fields available for editing will include Line, Type, Vial, Data file, Method and Sample Name.

8.6.4.1 Line refers to the sequential order of the sample log.

8.6.4.2 Type refers to the type of sample being run (sample, CCV, CCB, Calibration, etc.).

8.6.4.3 Vial is used to indicate the purge position on the autosampler.

8.6.4.4 Data file must be a unique name used for the storage of that file.

8.6.4.5 Method refers to stored parameters used for the acquisition, processing and reporting of the sample.

8.6.4.6 Sample name is a descriptor used in the reporting of the sample.

8.6.4.7 After sample log table is completed, select RUN and RUN SEQUENCE. The program will now download the GC, MS and data parameters for the sample on line 1 of the sample log.

8.7 Initiating a sequence for the purge-and-trap:

8.7.1 Using the keypad, located on the front of the PTA30/WS, press MANUAL MODE, and PROGRAM SET-UP. Use the arrow key to select AUTO and press PROGRAM SET-UP again. The operating parameters will appear individually each time PROGRAM SET-UP is pressed.

8.7.2 Enter the autosampler conditions specified in A. Instrument Conditions.

8.7.3 When conditions all entered, return to the screen which reads MAN<<AUTO>> and press AUTO MODE to begin the sampling sequence.

9.0 Qualitative Analysis

9.1 Target Compounds: Calibrated target compounds may be identified as present if the following criteria are met:

9.1.1 The intensities of characteristic ions of a compound should show symmetry and maxima in the same scan. Excessively noisy chromatograms may interfere with one or more ions in some cases.

9.1.2 The RRT of the sample component is within $\pm$0.06 RRT units of the RRT of the standard component.

9.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum..

10.0 Quantitative Analysis

10.1 When a compound has been qualitatively identified using its primary characteristic ion and its secondary ions (see Table 3), it can be quantitated using the integrated abundance of the primary characteristic ion.

10.1.1 All compounds are quantitated using the internal standard technique (see CALCULATIONS, section 11.0).

10.1.2 All manual integrations must be initialed and dated by the analyst.

10.1.3 The first order linear regression equation from the initial calibration is used to calculate the concentration in the sample.

10.2 Gasoline Range Organics (GRO) quantitation is based on a direct comparison of the total ion area within the retention time window to the total ion area of the Unleaded Gasoline Composite standard.

10.2.1 The total area is manually integrated with subtraction of Internal Standards and Surrogates present within the retention time window.

10.2.2 The retention time window is defined as beginning 0.1 min. prior to the onset of the methyl-tertiary-butylether peak and ending 0.1 min. after the conclusion of the napthalene peak (see Table 4).

10.2.3 The GRO concentration is calculated using the external standard method using formulas found in the CALCULATIONS section of this method.

11.0 Calculations

11.1 Relative Response Factor (RRF):

$$RRF = (Ax)(Cis)/(Ais)(Cx)$$

where:
Ax = area of compound quantitation ion
Ais = area of IS quantitation ion
Cis = IS concentration
Cx = compound concentration

An average RRF is calculated for each compound and surrogate from the initial calibration.

11.2 Percent Relative Standard Deviation:

$$\%RSD = \frac{\text{standard deviation}}{\text{average RF}} \times 100$$

where: average RF = average of five initial Response Factors for a compound

11.3 Percent Difference (%D):

$$\%D = \frac{(\text{average } RRF_i) - (RRF_c)}{(\text{average } RRF_i)} \times 100$$

where: average RRFi= average RRF from the initial calibration
RRFc = RRF from the continuing calibration standard

11.4 Target Compound Concentrations:

$$C_x = \frac{A_x C_{is} DF}{A_{is} RRF}$$

where: C_x = concentration of the compound in the sample
A_x = Area response of the characteristic ion for the compound to be measured
A_{is} = Area response of the characteristic ion for the internal standard
C_{is} = Concentration of the internal standard
DF = Dilution factor
RRF= Relative Response Factor

11.5 SMC Percent Recovery:

$$\%\ \text{Recovery} = \frac{(\text{concentration found})}{(\text{concentration spiked})} \times 100$$

11.6 Adjusted Practical Quantitation Limit for Samples:

$$\text{Adjusted PQL} = \frac{(PQL)}{D} \times DF$$

where: D = (100 - %moisture)
DF = Dilution Factor

11.7 Gasoline Range Organics Concentrations:

11.7.1 Determination of slope (m_{tph}):

$$m_{tph} = \frac{\sum (xi - \bar{x})(yi - \bar{y})}{\sum (xi - \bar{x})^2}$$

where: x_i = concentration of the standard
$\bar{x}$ = mean value of compound concentrations
y_i = individual response from the curve less internal standard and surrogates responses
$\bar{y}$ = mean value of all calibration responses

11.7.2 Determination of intercept (b):

$$b = \bar{y} - m_{tph}\,\bar{x}$$

where: $\bar{x}$ = mean value of compound concentrations
$\bar{y}$ = mean value of all calibration responses
m_{tph} = slope

11.7.3 Determination of GRO concentration in sample (C_{tph}):

$$C_{tph} = \left(\frac{R_s}{m_{tph}} - b\right)(D)$$

where: m_{tph} = slope
b = intercept of the calibration curve
R_s = GC response of sample in the GRO retention time window
D = dilution factor

12.0 Quality Control

12.1 Initial Calibration

12.1.1 The average RF must be calculated and recorded for each compound using the five RF values calculated from the initial calibration curve. The RSD for the calculated RFs should be ±20% for the initial calibration to be valid.

12.1.2 Using the responses and concentrations from the initial calibration, calculate and record the slope, correlation coefficient and intercept for GRO standards. The correlation coefficient should be greater than 0.995 for the calibration to be considered linear. If a correlation coefficient of less than 0.995 is found corrective action to eliminate the cause must be performed and documented prior to reattempting calibration.

12.1.3 Prior to the analysis of samples a method detection limit (MDL) must be performed to determine the minimum concentration of GRO that can be measured with 99% confidence that the value is above zero. Seven replicates of a standard prepared at 2-5 times the expected MDL are analyzed. The standard deviation of the seven values is calculated and multiplied by 3.14 to determine

the MDL.

12.2. Daily Continuing Verification

12.2.1 Prior to the analysis of any samples, inject 50 ng of 4-bromoflurobenzene (BFB) to check performance of the MSD. The resultant mass spectra for the BFB must meet all the criteria given in Table 1. These criteria must be demonstrated each 12 hour shift.

TABLE 1

BFB MASS - INTENSITY SPECIFICATIONS (4-Bromofluorobenzene)

Mass	Intensity Required (relative abundance)
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

12.2.2 The initial calibration curve for the GRO mix must be checked and verified once every 24 hours by analyzing a calibration standard which is at a concentration near the midpoint concentration for the working range of the GC/MS. The GRO mix must be within 80% -120% of the expected concentration for the initial calibration to be considered valid.

12.2.3 A method blank must be analyzed and shown to contain less than or equal to the PQL. If a method blank exceeds the limits for contamination, the laboratory shall investigate the source of contamination. Appropriate corrective action shall be taken and documented before further sample analysis occurs.

12.2.4 A matrix spike (MS) and matrix spike duplicate (MSD) should be performed for each batch of up to 20 samples analyzed by this method. The MS/MSD consist of 20 μL of the GRO mix spiked into 100 mL of sample (conc. = 500 ppb). Recovery windows and % difference limits are generated by the laboratory through analysis over time according to SW.846, Method 8000, sec. 80.

12.3 Samples

12.3.1 Surrogate recovery limits specified in Table 2 must be met for all blanks and samples. Surrogate recovery limits must be developed semi-annually.

TABLE 2

SURROGATE RECOVERY LIMITS - Water

Compound	Water % Recovery
Dichloroethane-d4	70-140
Toluene-d8	83-117
4-Bromofluorobenzene	64-132

12.3.2 Internal standard limits must be met for all blanks, samples, and MS/MSD. The IS area for the samples must be between -50 and +100% of the area in the mid-point standard calibration level. The RT shift must be between ± 30 seconds from the RT in the mid-point standard calibration level.

TABLE 3

Characteristic Masses (M/Z) for Target Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion
Fluorobenzene-IS	96	77
MTBE	73	57
Dichloroethane-d4-S.C.	102	
Toluene d8 -S.C.	98	
1,2-Dichlorobenzene d4-IS	152	
4-Bromofluorobenzene-S.C.	95	174,176
Napthalene	128	

TABLE 4

Characteristic Retention Times (minutes) and Method Detection Limits for Target Compounds

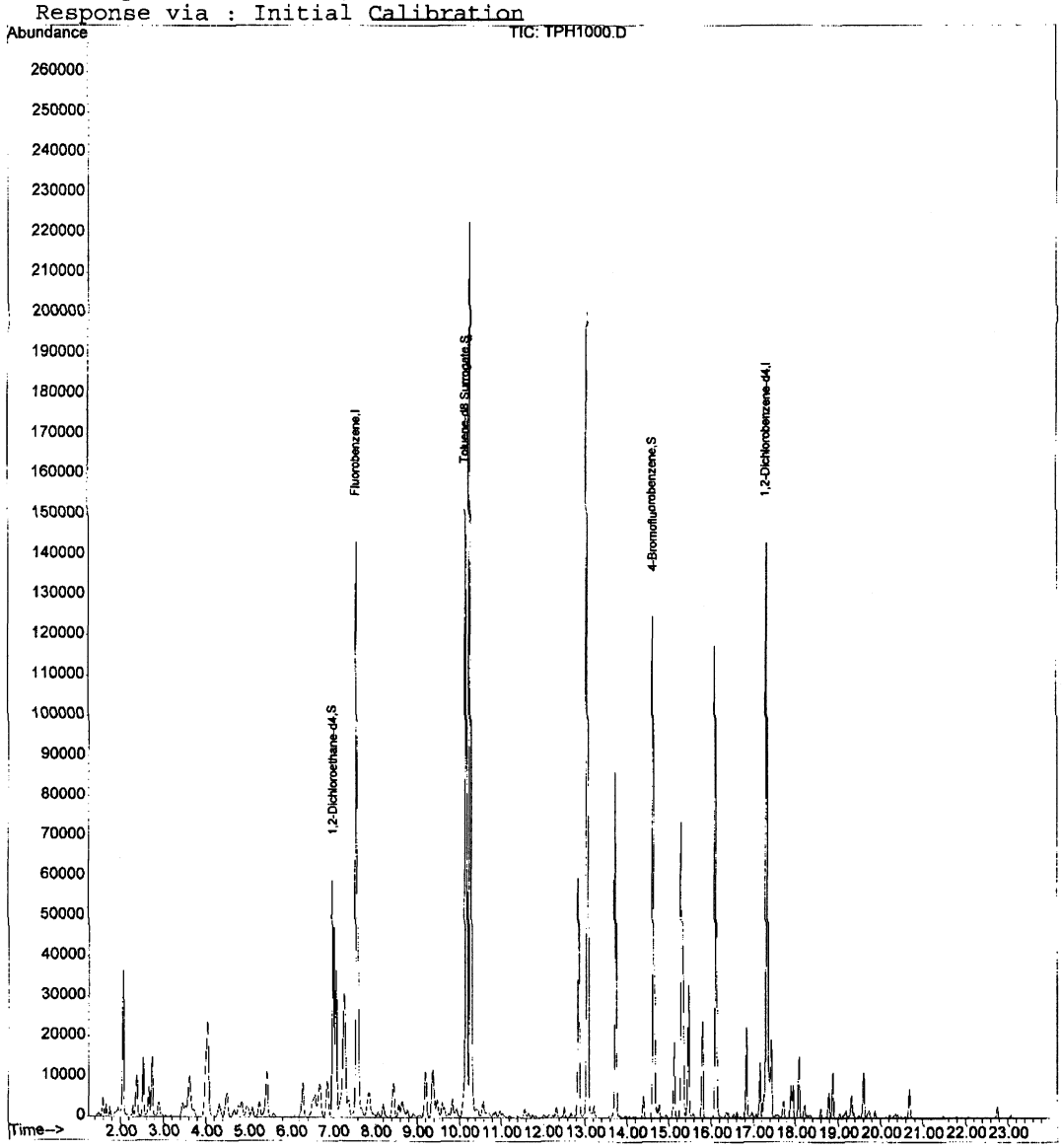
Compound	Retention Time	MDL(µg/L)
MTBE	4.04	5
Naphthalene	21.09	5
Fluorobenzene (IS)	7.80	
1,2-Dichlorobenzene D4 (IS)	17.56	
1,2-Dichloroethane-d4 (S)	7.23	
Toluene-d8 (S)	10.40	
4-Bromofluorobenzene (S)	14.87	

GAS CHROMATOGRAM OF GASOLINE STANDARD

Quantitation Report

Data File : D:\HPCHEM\1\DATA\051900\TPH1000.D Vial: 21
Acq On : 20 May 2000 1:53 am Operator:
Sample : 1000ppb GRO Inst : MS1
Misc : Multiplr: 1.00
MS Integration Params: LOWSENS.P
Quant Time: May 22 8:08 2000 Quant Results File: 8260TPH.RES

Method : D:\HPCHEM\1\METHODS\8260TPH.M (RTE Integrator)
Title : Method 8260
Last Update : Mon May 22 08:10:46 2000
Response via : Initial Calibration



TPH1000.D 8260TPH.M Wed May 24 08:22:49 2000 MS1 Page 2

Signature Page

To the best of my knowledge all information contained in this document is complete and accurate.

Author: _____ Date: 6-14-07
Kent Lawrence

Supervisor: _____ Date: 6-14-07
Kent Lawrence

Laboratory Director: _____ Date: 6/14/07
Michael Jaeger

QA Approval: _____ Date: June 14, 2007
Linda Felcyn

ARCADIS BBL

Attachment 1-3

KAR Wet Chemistry and
Miscellaneous Analysis SOPs

STANDARD OPERATING PROCEDURE

The Gravimetric Determination of Total, Fixed and Volatile Suspended Solids

1.0 References

1.1 EPA's *Methods for Chemical Analysis of Water and Wastes*, Method 160.2.

1.2 *Standard Methods for the Examination of Water and Wastewater*, 19th Edition, Method 2540 D.

2.0 Application

2.1 This method is applicable to all filterable aqueous samples.

3.0 Discussion

3.1 Total suspended solids content (TSS) is defined as the residue of sample retained by a 1.5µ glass fiber filter and dried to constant weight at 103-105° C. Fixed suspended solids (FSS) content is the residue of sample remaining after the TSS residue is ignited at 550° C. Volatile suspended solids (VSS) content is the portion of TSS residue lost after ignition at 550° C. The total suspended solids represent the sum of the fixed and volatile suspended solids.

3.2 When performing the fixed and volatile suspended analysis on a sample, the filter used for the total suspended solids analysis must first be ignited at 550° C in the muffle furnace.

4.0 Summary of Method

4.1 If pre-rinsed, pre-weighed filters are not used, a group of glass fiber filters is prepared. If fixed or volatile suspended analysis is to be performed, the filter is ignited at 550° C before being cooled in a desiccator. An aliquot of sample is filtered through the filter such that the residue after drying will be greater than 2.0 mg but less than 200 mg and will make it through the filter within a ten-minute period of time. The filter and residue are rinsed with deionized water and dried to constant weight at 103-105° C. If fixed or volatile analysis is required, the dried residue is ignited at 550° C to constant weight. The various results are calculated from the mass of each residue and the volume of sample analyzed.

5.0 Sample Handling

5.1 Collect a minimum of 500 mL sample in plastic. Perform the analysis as soon as possible if the sample is not refrigerated; within seven days if refrigerated at ~4° C.

6.0 Interferences

6.1 The sample must be thoroughly mixed before removing a representative subsample. Large non-homogeneous particles generally should not be included in a sample aliquot unless the analyst feels they are representative of the wastestream.

6.2 Avoid creating large, water-trapping crusts on the residue by limiting the sample volume to a residue of 200 mg or less.

6.3 All suspended solids should be thoroughly rinsed after filtration to remove any dissolved solids which may create a positive interference.

6.4 Excessively long filtering times (>10 minutes) are an indication that the filter is plugging, a source of positive interferences.

6.5 The desiccator in which the filter/pan assembly is stored throughout the analysis must achieve and maintain a relative humidity of 20% or lower.

6.6 The temperature of the ovens must be monitored daily using a calibrated thermometer to ensure the proper temperature is being maintained.

6.7 The desiccators must be monitored daily and recharged as needed in order to provide a dry (relative humidity less than 20%) environment for cooling of residues.

6.8 Always handle the glass fiber filters using the angled, paddle-type forceps. Damaged filters are more prone to loss of mass during the analysis.

6.9 When pre-weighed filters are used, the pan is not part of the tare weight so the filter must be removed from its pan. If the filter is bumped or dropped during transfer to and from its pan, a negative loss is likely and the analysis must be repeated. Keep the filter horizontal during transfers and handle with extreme care.

7.0 Equipment

7.1 Pre-rinsed, pre-weighed filters
Whatman 934-AH glass fiber filters, 1.5µ, 47 mm diameter
Disposable aluminum weigh pans, to hold 47 mm filter
Crucible, large enough to accommodate filter(s) used
Vacuum filtration assembly
Graduated cylinders, glass, Class A

Transfer pipet, 25 mL, wide-aperture
Gravity oven, capable of maintaining 103-105° C
Muffle furnace capable of maintaining 550° C
Forceps, angled, paddle-type
Crucible tongs
Analytical balance with 0.1 mg capability
Desiccator, properly charged

8.0 Chemicals and Reagents

8.1 Desiccant, $CaSO_4$, mixed with color indicator, purchase fresh

9.0 Safety

9.1 Do not touch hot parts of the oven or furnace, or any items recently removed from them. Use crucible tongs to handle hot dishes.

9.2 Wear rubber gloves and wash hands frequently when handling wastewater samples.

10.0 Procedure

10.1 Charge the desiccator with fresh desiccant. The desiccator must achieve and maintain a relative humidity of 20% or less before use. Monitor desiccators daily when in use.

10.2 Filter Preparation

10.2.1 Label a sufficient number of aluminum weigh pans using a permanent marker.

10.2.2 Rinse each filter by placing it, wrinkled side up, on the magnetic filtering apparatus. Fill the assembly with 250 mL of deionized water and apply vacuum. When the volume of water has drawn down, rinse the filter with a direct stream of deionized water. Transfer the filter to a labeled aluminum weigh pan and dry thoroughly in an oven at 103-105°C for at least 6 hours.

10.2.3 If a fixed or volatile analysis is required, transfer the dry filter from the oven to a large crucible and ignite in a muffle furnace at 550°C for 20 minutes. Cool the crucible to 103-105° C before transferring the ignited filters to their labeled pans.

10.2.4 Transfer each filter/pan set to a desiccator to cool for at least 15 minutes.

10.2.5 For pre-rinsed, pre-weighed filters, record in the Suspended Solids notebook the pan ID and tare weight found on the pan. If necessary, ignite the filter as in 10.2.3.

10.3 Sample Analysis

10.3.1 Ensure the analytical balance is properly calibrated and record the mass of each filter/pan set to 0.1 mg.

10.3.2 Carefully remove a filter from its pan using the angled paddled forceps and place it on the filtering assembly. Seat the filter in the assembly by wetting the filter with a small quantity of deionized water and applying the vacuum.

10.3.3 Estimate visually the volume of sample needed to produce a residue with a mass greater than 2 mg but less than 200 mg. It is sometimes helpful to refer to past data obtained on the wastestream; however, wastestreams can change, and past results are not always the best indication of the optimal sample volume for the analysis at hand.

10.3.4 Shake the sample vigorously to suspend any settled solids, quickly remove the lid and pour off a representative aliquot into a graduated cylinder. A minimum of 5.0 mL of sample must be used for samples with heavy solids; a minimum of 250 mL must be used for the standard reporting limit of 4 mg/L. Lower detection limits can be achieved by filtering a larger volume of sample.

NOTE: Samples with extremely heavy solids may need to be stirred using a stir plate and magnetic stir bar in a 1 L open-cup beaker. In such cases, stir the sample so that the vortex extends one-third of the way into the sample, place the tip of a wide-aperture transfer pipet half-way into the sample and half-way between the center of the vortex and the wall of the beaker, and draw up an appropriate volume of sample.

10.3.5 Transfer the sample aliquot to the filter mounted on the filtering apparatus and apply the vacuum. Rinse the cylinder or pipet thoroughly with deionized water to transfer all of the solids to the filter. If less than 250 mL sample was used and the sample was drawn through very rapidly (less than 10 seconds), reshake the sample and transfer another aliquot of sample. If the sample takes more than 10 minutes to filter, start again at 10.3.2 using less sample and a new, tared filter/pan set.

NOTE: If the minimum of 5 mL of sample cannot be successfully filtered and rinsed within the ten-minute period, the analyst may either 1) report an inconclusive result or 2) analyze a dilution of the sample and report a qualified estimate. Ask Client Services to consult the client for guidance.

10.3.6 Record the sample ID and volume of sample associated with each filter/pan set.

10.3.7 Rinse the solids and the filter with a gentle but direct stream of deionized water, allowing complete drainage through the filter with each rinse.

Maintain the vacuum for 10-15 seconds after the final rinse. Carefully remove the filter from the housing, return it to its pan and dry in an oven at 103-105° C for at least four hours.

10.3.8 Remove the set from the oven and let cool in the desiccator for 15 minutes. Check the calibration of the balance and record the mass of each filter or filter/pan set, depending on whether pre-weighed filters were used. Return each set to the oven for at least one more hour of drying. Let cool, weigh and record the mass. Repeat this procedure until constant weight is achieved.

NOTE: Constant weight has been achieved when the difference in mass between two separate weighings (with at least one hour of additional drying in between) is 0.5 mg or less.

10.3.9 If fixed and/or volatile suspended solids are to be determined, remove the dried filter with residue, place it in the crucible and ignite to constant weight at 550° C. Record the mass of the ignited residue and filter after proper cooling.

11.0 Calculations

11.1 Total suspended solids $= \frac{(B - A) \times 1000}{\text{sample volume, L}}$ (mg/L)

Fixed suspended solids $= \frac{(C - A) \times 1000}{\text{sample volume, L}}$ (mg/L)

or

$= \frac{(C - A)}{(B - A)} \times 100$ (% of total solids)

Volatile suspended solids $= \frac{(B - C) \times 1000}{\text{sample volume, L}}$ (mg/L)

or

$= \frac{(B - C)}{(B - A)} \times 100$ (% of total solids)

where, A = mass of filter, g
B = mass of dried filter and residue, g
C = mass of ignited filter and residue, g

NOTE: When pre-weighed filters are not used, the mass values for A, B and C will include the mass of the pan.

12.0 QA/QC

12.1 Perform one blank analysis every ten samples. If the blank residue is greater than ±0.5 mg of the initial tare weight, report no results and reanalyze the entire batch.

12.2 Perform one duplicate analysis every ten samples. If the relative percent difference between duplicate analyses is greater than 2 standard deviations from the statistical mean (LIMS dup graph), the analyst should investigate all sources of error and determine if the QC failure is due to a matrix effect before reporting any results. Qualify approximate results with the appropriate code.

13.0 Reporting

13.1 Report results to the 1 mg/L unit with no more than three significant figures.

13.2 The limit of detection for a 250 mL sample analysis will be 4 mg/L, which is based on a raw residue mass of at least 1.0 mg. A limit of detection of 1 mg/L may be reported if a 1000 mL sample is filtered and has a filter residue of less than 1.0 mg.

13.3 If an inconclusive result was obtained (section 10.3.5), report *See comment* in the LIMS Concentration field and enter the IR code. If the sample was diluted and an estimated result reported, qualify the result with the AP code.

13.4 If a non-detect result was obtained for the TSS analysis, then 1) report the same non-detect result for an accompanying FSS or VSS order, 2) apply the TA code to the FSS and/or VSS result, and 3) enter XXX in the analyst initials field for the FSS and/or VSS result.

Signature Page

To the best of my knowledge all information contained in this document is complete and accurate.

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Dave Alkema

Supervisor: Michael Jaeger Date: 5/25/06
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Laboratory Director: Michael Jaeger Date: 5/25/06
Michael Jaeger

QA Approval: Pollyann Nee Date: 30-May-2006
Pollyann Nee

SOP No.: KQ450.05
Date Issued: June 28, 2006
Next Revision Due: 06/08

STANDARD OPERATING PROCEDURE

The Colorimetric Determination of Various Forms of Phosphorus

1.0 References

1.1 *Methods for Chemical Analysis of Water and Wastes*, U.S. EPA, Method 365.2.

1.2 *Standard Methods for the Examination of Water and Wastewater*, 19th Ed., Method 4500-P sections A,B,E.

2.0 Application

2.1 This SOP can be applied to aqueous samples, extracts, filtrates, sludges and, in some cases, solids. Samples requiring only orthophosphate analysis must be relatively free of any turbidity or interfering color. To perform a total phosphorus digestion on samples which are solid or contain a significant quantity of solids, the solids must be either water-soluble or fine enough to be suspended in water for diluting purposes.

3.0 Discussion

3.1 Because phosphorus is an essential element for all organisms to live and grow, the accurate quantitation of various forms of phosphorus is of interest to many different industries; most notably, the water quality industry. Phosphorus is found in waters, wastewaters and wastewater residues almost exclusively as phosphates. These consist of orthophosphate, condensed phosphates (pyro-, meta- and polyphosphates), and organically-bound phosphorus. These forms of phosphorus make their way into waters and wastewaters from a variety of sources such as water treatment chemicals, agricultural fertilizers, domestic sewage and detergents.

3.2 This SOP contains procedures for determining orthophosphate, acid-hydrolyzable phosphorus, organic phosphorus and total phosphorus. The orthophosphate determination is not actually specific for orthophosphate exclusively; a small amount of weakly condensed phosphates will react with the color reagent. The result of direct color development is sometimes referred to as reactive phosphorus, but KAR regards the result as a measure of orthophosphate. Similarly, the acid hydrolysis for condensed phosphates will reduce some organic phosphorus to the reactive (orthophosphate) form. The thermal, oxidative digestion for total

phosphorus generally converts all condensed and organic phosphorus into orthophosphate for color development. Always bring orthophosphate standards through the acid hydrolysis or total digestion when those treatments are required.

3.3 KAR's Dissolved Phosphorus analysis is understood to be a measure of dissolved orthophosphate. The sample should be analyzed as soon as possible after collection and not preserved. An unfiltered, preserved sample cannot be later filtered and analyzed for dissolved phosphorus.

4.0 Summary of Method

4.1 For the acid hydrolysis and total determinations, a set of orthophosphate calibration standards are digested with the sample at 120°C after addition of the appropriate digestion solution. After cooling, the orthophosphate is color-developed using an ascorbic acid/molybdate reagent; the absorbance is measured spectrophotometrically at 880 nm. Organic phosphorus and acid-hydrolyzable phosphorus are obtained by calculation.

4.2 For the orthophosphate analysis, the sample is filtered and/or prepped with a color blank (if necessary), color-developed and analyzed spectrophotometrically.

5.0 Sample Handling

5.1 A 100 mL portion of sample is collected in glass or plastic free of phosphorus contamination. The orthophosphate analysis is performed on the unpreserved sample within 48 hours of collection. The total and acid-hydrolyzable analyses are performed within 28 days of collection on a sample preserved to pH < 2 with sulfuric acid and refrigerated.

6.0 Interferences

6.1 Since most detergents contain some phosphorus, any glassware or containers used in this analysis which have been cleaned with detergent will almost certainly give a positive interference. See Cleaning Procedure.

6.2 Various filters that may be used for filtering either the sample or an extract may contain significant amounts of orthophosphate, which will cause a positive interference. Always prepare a filter blank when filtering.

6.3 Arsenates will react with the molybdate reagent in a similar fashion to phosphate to give a positive interference. Hexavalent chromium and nitrite give a negative interference in high concentrations. Any turbidity still present in a digested sample will give a positive interference. A turbid digest must be filtered prior to color development.

7.0 Equipment

7.1 Spectrophotometer, at 880 nm
Calibrated vials, plastic, 15 mL (Evergreen Sci. P/N 290-3615-060)
Digestion Block and hotplate, at 120° C
Boiling chips
Syringes, disposable
Syringe filters, 0.45µ, acid-rinsed

8.0 Chemicals and Reagents

8.1 Hydrochloric acid, HCl
Potassium phosphate, monobasic, KH_2PO_4 (potassium dihydrogen phosphate)
Ammonium persulfate, $(NH_4)_2S_2O_8$
Sulfuric acid, H_2SO_4
Ammonium molybdate, $(NH_4)_2Mo_2O_7 \cdot 4H_2O$
Potassium antimonyl tartrate, $Sb_2C_8H_4O_{12}K_2 \cdot 3H_2O$
Ascorbic acid, $C_6H_8O_6$
Nitric acid, HNO_3
Sodium hydroxide, pellets, NaOH
Phenolphthalein, disodium salt

8.2 Preparation of Reagents

NOTE: Any glassware and labware used to prepare the following reagents must be free of any phosphorus contamination. See Cleaning Procedure.

8.2.1 Hydrochloric acid, 1%

In a fume hood, carefully add 10 mL of conc. HCl to 1000 mL of deionized water. Store at room temperature in a dedicated 1 L plastic container. Prepare fresh every six months.

8.2.2 Digestion Solution

Dissolve 32.5 g of ammonium persulfate into approximately 800 mL of deionized water in a 1L volumetric flask container. Add 12.5mL of conc. sulfuric acid, dilute to the mark and mix well. Store at room temperature in plastic. Prepare fresh every six months.

8.2.3 Sulfuric Acid, 5N

Dilute 70 mL of conc. H_2SO_4 carefully to 500 mL of deionized water. Store in plastic at room temperature. Prepare fresh every six months.

8.2.4 Ammonium Molybdate (AM) Solution

Dissolve 10 g of ammonium molybdate into 250 mL of deionized water. Store in plastic at room temperature. Prepare fresh every six weeks.

8.2.5 Potassium Antimonyl Tartrate (PAT) Solution

Dissolve 1.3714 g of potassium antimonyl tartrate into 500 mL of deionized water. Store in plastic at room temperature. Prepare fresh every six months.

8.2.6 Ascorbic acid, 0.1M

Dissolve 1.76 g of ascorbic acid into 100 mL of deionized water. Store in plastic or glass and refrigerate. Prepare fresh every 2 weeks.

8.2.7 Color Reagent

Mix the reagents in the following proportion and order in a disposable beaker , swirling after each addition (this reagent is stable for 4 hours):

20 mL 5N H_2SO_4
2 mL Potassium Antimony Tartrate Solution
6 mL Ammonium Molybdate Solution
12 mL Ascorbic Acid Solution

8.2.8 Acid Hydrolysis Solution

Add 30 mL of conc. H_2SO_4 and 0.4 mL conc. HNO_3 to 60 mL deionized water in a 100 mL volumetric flask. Dilute to the mark and mix well. Store in plastic at room temperature. Prepare fresh weekly.

8.2.9 Sodium Hydroxide Solution, 6 N

Dissolve 24.0 g of NaOH (pellets) in 90 mL deionized water in a 100 mL volumetric flask. Allow to cool. Dilute to volume, mix well and store in plastic at room temperature. Prepare fresh every six months.

8.2.10 Phenolphthalein Indicator Reagent (Aqueous)

Dissolve 0.5 g of Phenolphthalein disodium salt in 100 mL deionized water. Store in plastic at room temperature. Prepare fresh every six months.

9.0 Safety

9.1 Sulfuric acid and nitric acid are highly corrosive mineral acids. Use extreme care when handling. Wear safety glasses, gloves and a labcoat to prevent contact with skin when handling. Wipe up all small spills immediately with water.

9.2 Potassium antimony tartrate and ammonium molybdate are toxic. Do not breathe dust and avoid dermal contact.

9.3 Ammonium persulfate is highly reactive and is a strong oxidizer. Keep away from materials that are easily reduced, especially organics. Wear dermal protection when handling it.

9.4 Hydrochloric acid is a highly corrosive, volatile acid. Always handle in a fume hood with safety protection.

10.0 Preparation of Standards

NOTE: All glassware and labware used to prepare the following standards must be free of all phosphorus contamination. See Cleaning Procedure (section 11.1).

10.1 Phosphorus Stock Standard, 50.0 mg/L (as P)

Dry approx. 150 mg KH_2PO_4 in an oven at 104° C for 24 hours. After cooling in a desiccator, dissolve 109.7 mg in a 500 mL volumetric flask containing 400 mL deionized water, dilute to mark and mix well. Store in plastic at room temperature with one drop of conc. sulfuric acid to preserve. Prepare fresh every six months.

10.2 Phosphorus Stock Standard, 1.0 mg/L (as P)

Pipet 10.0 mL of the 50 mg/L stock in a 500 mL vol. flask containing 400 mL of deionized water. Dilute to mark and mix well. Store in plastic at room temperature with one drop of conc. sulfuric acid to preserve. Prepare fresh every six months.

10.3 Calibration Standards

Prepare a set of 0, 0.02, 0.2 and 1.2 mg/L standards in the following manner: For the 0 mg/L standard, place 5.0 mL of deionized water into a sample vial. For the 0.02, 0.2 and 1.2 mg/L standards, pipet 0.10, 1.0, and 6.0 mL of the 1.0 mg/L standard respectively into their own 15 mL vial. Dilute the 0.02 and 0.2 mg/L standards up to the 5.0 mL mark with deionized water.

10.4 Spiking standard, 20.0 mg/L (as P)

Dry 1000 mg of KH_2PO_4 in an oven at 104° C for 24 hours. After cooling in a desiccator, place 878 mg into a 1 L volumetric flask and dilute to the mark. Place 10.0 mL of this solution into a 100 mL volumetric flask, dilute to the mark and

mix well. Store in plastic in the dark at room temperature with one drop of concentrated sulfuric acid to preserve. Prepare fresh every six months. A 50 µL spike of this solution to 5.0 mL of sample represents a 0.20 mg/L addition of phosphorus.

10.5 Check Standard(s)

Purchase a certified standard and prepare a working standard in the 0.1-1.0 mg/L range. Record the certified (prepared) value and the 95% confidence limits on the container. Store an orthophosphate check standard in plastic in a refrigerator. Store a total phosphorus check standard in plastic at room temperature. Prepare fresh every six months. Prepare a check standard for analysis by pipetting 5.0 mL into its own 15 mL vial.

11.0 Procedure

11.1 Cleaning Procedure

11.1.1 Thoroughly rinse all vials (not the caps) with dilute 1% HCl. Rinse with deionized water three times. It is helpful to prep a large number of vials in advance.

11.2 Total Analysis Digestion and Prep

11.2.1 Preheat the digestion block to 120° C.

11.2.2 Prepare a set of calibration standards and transfer 5.0 mL of sample (or dilution) to its own vial.

11.2.3 To all of the tubes, add 2.0 mL of the digestion solution and add a single boiling chip. Place all the tubes into the digestion block with the caps <u>loosely</u> threaded onto the tubes.

11.2.4 If the block is preheated, the tubes should come to a boil within a few minutes. Maintain a slow boil for one hour.

11.2.5 Remove the tubes from the digestion block and let them cool at least 10 minutes before bringing the volume of each tube up to 12 mL using deionized water. Filter any digestates that contain turbidity. At this point, the tubes can go straight to color development or be capped and saved at room temperature overnight.

11.3 Orthophosphate Prep

11.3.1 For orthophosphate analysis, transfer 5.0 mL of sample (or an appropriate dilution) to its own labeled vial. All samples must be filtered. Prepare a

duplicate prep as a color blank for any sample that is not visibly clear and colorless.

11.3.2 Bring each tube up to the 12mL mark with deionized water. The tubes are now ready for color development.

11.4 Acid Hydrolysis Prep

11.4.1 To each sample and standard, add one drop of phenolphthalein indicator and mix. Add Acid Hydrolysis Solution dropwise to each prep so that the pink color is just discharged. Then add 3 drops more.

11.4.2 Add a boiling chip and boil gently for 3 hours with the lids loosely covering the tubes. Allow to cool.

11.4.3 Neutralize each prep to a faint pink color with a dropwise addition of 6 N NaOH. Bring each tube up to the 12 mL mark with deionized water. The tubes are now ready for color development (11.5).

11.5 Color Development and Analysis

11.5.1 Set the wavelength of the spectrophotometer at 880 nm, turn it on and let it warm up for at least 15 minutes.

11.5.2 Add 1.2 mL of the ascorbic color reagent to each of the tubes and mix each tube thoroughly.

11.5.3 After 10 minutes but no longer than 25 minutes, zero the spectrophotometer using the 0 mg/L standard. Measure and record the absorbance of each standard and sample at 880 nm using the 1" cuvette.

11.5.4 Samples which produce an absorbance reading higher then the 1.2 mg/L standard must be redigested using a smaller amount of sample.

12.0 Calculations

12.1 Construct a calibration curve of absorbance vs. concentration using a statistical calculator. Convert sample absorbances to results in mg/L. Apply any dilution factors.

12.2 Unless otherwise requested by the client, all forms of phosphorus should be reported "as P." If a client requests that phosphorus be reported "as PO_4", multiply the non-rounded "as P" result by 3.066 and then perform rounding.

12.3 Determine "Acid-hydrolyzable Phosphorus" by subtracting the Orthophosphate result from the result obtained after acid hydrolysis. Determine Organic Phosphorus by subtracting the result obtained after acid hydrolysis from the result obtained after the total digestion.

13.0 QA/QC

13.1 Perform a lab method blank analysis every analytical batch and every ten samples. If the result is greater than 0.02 mg/L, investigate all potential sources of contamination and reanalyze any samples with results that are suspect due to the failed blank result. Correct any problem before reanalyzing.

13.2 Perform a matrix spike analysis every analytical batch and every ten samples. If the spike recovery falls outside the control limits charted by LIMS, investigate all potential sources of error and correct any problem before reporting results. Qualify any result that fails due to a matrix effect.

13.3 Perform a duplicate analysis every analytical batch and every ten samples. If the relative percent difference falls outside the control limit charted by LIMS, investigate all potential sources of error and correct any problem before reporting results. Qualify any result that fails due to a matrix effect.

13.4 Perform a check standard analysis every analytical batch. If the recovery falls outside the 95% confidence limits indicated by the manufacturer, investigate all potential sources of error and reanalyze any samples with results that are suspect due to the failed check standard recovery. Correct any problem before reanalyzing.

14.0 Reporting

14.1 Unless requested otherwise, report results "as P" in mg/L to the 0.01 unit with no more than three significant figures. The detection limit for phosphorus using this method is 0.02 mg/L for aqueous samples. The LOD for a soil, sludge or waste material will depend upon the amount of sample digested.

Signature Page

To the best of my knowledge all information contained in this document is complete and accurate.

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Dave Alkema

Supervisor: Michael Jaeger Date: 6/27/06
Michael Jaeger

Laboratory Director: Michael Jaeger Date: 6/27/06
Michael Jaeger

QA Approval: Jane M. Clark Date: 28-Jun-06
Jane Clark

STANDARD OPERATING PROCEDURE

Determination of Fluoride, Chloride, Nitrite, Nitrate, Bromide, Phosphate and/or Sulfate in Water by Ion Chromatography

1.0 References

1.1 *Training Manual: Lachat QuikChem 8000 Ion Chromatograph*, Zellweger/Lachat Instruments

1.2 *Test Method for the Determination of Inorganic Anions in Water by Ion Chromatography*, EPA Method 300.0A

1.3 *Determination of Inorganic Anions by Ion Chromatography*, EPA Method 9056

1.4 *Standard Methods for the Examination of Water and Wastewater*, Method 4110 B, "Determination of Anions by Ion Chromatography," 18th Edition.

2.0 Application

2.1 This SOP describes the determination of chloride, fluoride, bromide, nitrate, nitrite, phosphate, and/or sulfate in aqueous samples. The linear range concentrations for the various anions are as follows: 0.05-5.0 mg/L for fluoride, bromide, nitrite, nitrate and phosphate; 0.5-50 mg/L for chloride; and 1.0-100 mg/L for sulfate. Samples with concentrations higher than the upper limit may be brought in range by an appropriate dilution. The reporting limit or practical quantitation limit (PQL) for each anion is the lower limit of the ranges listed above.

3.0 Summary of Method

3.1 A small volume of sample is filtered, poured into a test tube, loaded onto an autosampler tray along with a set of calibration standards and requisite QC samples, and the complete batch submitted for ion chromatography analysis. The autosampler injects an aliquot of the sample into a stream of carbonate-bicarbonate eluent which passes through a guard column, separator column, suppressor column, and finally a conductivity detector.

3.2 Each anion is identified and quantitated based on a comparison of its retention time and peak area against a set of calibration standards. The software which runs the instrument performs all regression calculations and compiles a final report.

4.0 Discussion

4.1 Ion chromatography is a technique of chemically separating a mixture of ions in an aqueous matrix. This SOP applies only to the separation, detection and quantitation of the anions specified above. A small portion of sample containing the mixture of ions passes through two separation columns where the ions are separated into discrete bands based on their electronic affinity for the exchange sites of the column resin.

4.2 The ions, now separated, then pass through a suppressor column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids. The separated anions in their acid form are finally detected while passing through a flow-through conductivity cell.

4.3 The change in signal generated by each anion band passing through the conductivity cell creates a "peak" with its own retention time and measurable area. Calibration standards analyzed under the same conditions define a concentration range for the accurate identification and quantitation of the ions in the mixture.

5.0 Interferences

5.1 Any species with a retention time similar to that of the desired ion will interfere. High levels of some organic acids may interfere with this anion analysis. Two common species, formate and acetate, elute between fluoride and chloride. Large quantities of ions eluting close to the ion of interest will also result in an interference. Bromide and nitrate elute very close together and are potential interferences for each other. It is advisable not to have bromide/nitrate ratios higher than 1:10 or 10:1 if both anions are to be quantified.

5.2 If one anion is observed to interfere with another, the first approach should be to dilute the sample to such a point that the analyte of interest is still in the linear range but the interference has been overcome. If this is not successful, adopting a different integration scheme can sometimes help overcome interferences. It is essential that the sample peaks be integrated with the same scheme as the calibration standards. Occasionally, separation can be improved by adjusting the eluent concentration and/or flow rate.

5.3 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus. To prevent interferences to consistent flow, all samples require membrane filtration (0.45µm) prior to analysis. Prepare all standards, reagents and diluents using water and glassware that is known to be free of the analytes of interest. The routine analysis of blanks, internal check standards and external control standards is absolutely essential in monitoring for these systematic interferences.

6.0 Apparatus and Equipment

Ion chromatograph (Zellweger/Lachat QuikChem 8000) equipped with:
System Unit (Lachat #A83100)
IC/FIA Software (Lachat #A88001)
Sample Processing Module (Lachat #A84000)

Guard column, anions (Lachat #28085)
Analytical column, anions (Lachat #28084)
Suppressor column (Lachat #28097)
Conductivity module, CM-100 (Lachat #A28982)
Watson-Marlow (peristaltic) pump (Lachat #A82106)
Eluent pump (Lachat #A28103)
Autosampler, AIM 1250 (Lachat #A81100)
System computer w/ printer
Watson-Marlow pump cassette (Lachat #22121)
Rheodyne ferrule/nut (Lachat #28051)
Upchurch ferrule/nut (Lachat #28057)
Pump tube adapter w/ large collar (Lachat #50907)
Tube connector (Lachat #50009)
PEEK tubing, tan, 0.010" i.d. (Lachat #28031)
PEEK tubing, green, 0.032" i.d. (Lachat #28032)
PEEK tubing, yellow, 0.007" i.d. (Lachat #28052)
Flow fit union (Lachat #50901)
Connector (Lachat #50015A)
O-ring, Viton (Lachat #50013)
Pump tubing, white-white-white (Lachat #54309)
Pump tubing, black-black-black (Lachat #54307)
Pump tubing, orange-orange-orange (Lachat #54308)
PEEK tubing cutter (Lachat #28080); replacement blades (Lachat #28081)
Sample loop, 200 µL (Lachat #28055)
Syringe, 30 mL (Lachat #28120)
Eluent, 2½ L clean glass bottle, plastic-coated
Regenerant reservoir, 3 L plastic

Analytical balance, capable of weighing to 0.0001 g
Top-loading balance, capable of weighing to 0.001 g
Volumetric (Class A) pipets, flasks, beakers: assorted sizes

7.0 Preventive Maintenance

7.1 Perform the following maintenance as needed:
- Keep all external surfaces clean and dry
- Clean pump cassettes
- Clean and lubricate pump roller surfaces with silicon spray
- Clean autosampler rods
- Replace pump tubing
- Inspect ports, valves and ferrules

7.2 Clean column as needed.

7.2.1 Azide treatment: Prepare 250 mL 0.25 M sodium azide solution. Turn the columns around so that flow will be in the opposite direction of normal running. Run the azide solution through the system for 30 minutes at 0.5 mL/min and follow with a deionized water rinse.

7.2.2 Methanol treatment: Prepare a 5% methanol solution. Disconnect the green PEEK tubing connected to port 2 of the six-port valve. Place the eluent line in reagent water and prime the pump and PEEK tubing to prevent air getting on column. Connect the green PEEK tubing directly to the guard column inlet and set a small beaker at the guard column outlet to collect the waste. Set the eluent pump at 1.0 mL/min and pump 30 mL of reagent through the column. Stop the pump, disconnect the guard column from the green PEEK tubing, place the eluent line in the methanol solution, reverse the column and reconnect the green PEEK tubing to the guard column outlet. Pump 30 mL of methanol solution through the column at 1.0 mL/min, stop the pump, place the eluent line back in the reagent water, and pump reagent through the column at 1.0 mL/min for 30 minutes. Stop the pump, return all lines and connections to their normal configuration, place the eluent line back in the eluent, and run the system as usual.

7.2.3 Sodium EDTA treatment: Prepare 10 mL of 50 mM tetrasodium EDTA and pump through the columns under normal conditions. Do NOT contaminate eluent with EDTA solution; rinse inlet filter thoroughly with deionized before returning the line to the eluent reservoir.

7.3 Clean eluent lines every six months or as needed. Boiling water treatment: Boil one liter of deionized water. Disconnect columns from eluent lines and pump entire liter of boiling water through the lines at 2.0 mL/min (keep water boiling during pumping). Repeat with two more liters of boiling water.

7.4 Replace ferrules, filter inlet and guard disks every six months per training.

7.5 Replace Teflon and PEEK tubing every six months.

7.6 Clean up files on hard drive every six months or as needed per training.

8.0 Safety

8.1 Always wear safety glasses, labcoat and protective gloves when working with potentially hazardous materials and samples in the lab. Wash hands often.

8.2 Sodium azide and sodium fluoride are toxic. Do not inhale dust. Handle in a fume hood with dermal protection.

8.3 Boiling water can damage skin and should be handled with care with insulated protection. Always boil water in an annealed glass container.

8.4 Methanol is a volatile, flammable solvent. Store and handle away from open flames and sources of sparks. Make transfers in a fume hood.

8.5 Sulfuric acid is a highly corrosive mineral acid. Always handle with dermal protection away from strong bases. Keep neutralizing spill kits nearby while handling. Clean up small spills immediately with a large volume of water.

9.0 Preparation of Reagents

9.1 Reagent purity

9.1.1 Use analyte-free, deionized lab water (>10 megohm).

9.1.2 All chemicals used to prepare reagents must be Reagent Grade quality or better.

9.2 Sodium Azide Solution, 0.25M

Dissolve 4.06 g of sodium azide into 250 mL reagent water. Make up fresh as needed.

9.3 Methanol Solutions, 5% and 20%

For the 5% solution dilute 12.5 mL methanol to 250 mL with reagent water and mix well. For the 20% solution dilute 50 mL methanol to 250 mL with reagent water and mix well. Make up fresh as needed.

9.4 Sodium EDTA Solution, 50 mM

Dissolve 0.026 g tetrasodium EDTA in 250 mL of reagent water. Store in plastic container and refrigerate. Prepare fresh monthly.

9.5 Sodium Bicarbonate Stock, 100 mM $NaHCO_3$

Dissolve 4.20 g sodium bicarbonate volumetrically to 500 mL with reagent water. Store in glass and refrigerate. Make up fresh monthly.

9.6 Sodium Carbonate Stock, 100 mM Na_2CO_3

Dissolve 5.30 g sodium carbonate volumetrically to 500 mL with reagent water. Store in glass and refrigerate. Make up fresh monthly.

9.7 Daily Eluent, 2.0 mM $NaHCO_3$/2.6 mM Na_2CO_3

Pour approximately 30 mL each of the bicarbonate and carbonate stocks into separate plastic beakers to prevent contamination of the stock standards. Transfer 20 mL of the bicarbonate stock and 26 mL of the carbonate stock to a 1 L volumetric flask, dilute to the mark with reagent water and mix well by inversion (do not use a stir bar and stirrer). Degas the eluent just prior to use with a high-purity helium sparging for 1 min/L.

9.8 Suppressor regenerant solution, 0.25 M H_2SO_4

Transfer 14 mL of conc. H_2SO_4 to 500 mL reagent water and dilute to 1 L with reagent water. Store refrigerated in 1-gallon plastic container and make fresh every 6 months.

10.0 Preparation of Standards

10.1 Because the following standards are prepared gravimetrically, any errors in weighing cannot be corrected by volumetric dilution. Prepare all standards exactly as described.

All calibration standards are to be prepared fresh weekly unless nitrite, nitrate and phosphate are included; then the standards must be prepared daily.

10.2 Bromide stock solution (100 mg Br^-/L)

Purchase a certified 1000 ppm standard. Transfer 10.00 g of standard to tared 100 mL volumetric flask, dilute to 100.0 g with reagent water and mix well. Store certified standard in its own container and the working stock in a 125 mL plastic container in a refrigerator; prepare the working stock fresh every six months. Purchase a new certified standard annually.

10.3 Chloride stock solution (1000 mg Cl^-/L)

Purchase a certified standard. Alternatively, dry approximately 0.5 g of sodium chloride (NaCl) in an oven for 30 minutes at 105° C, and cool in a desiccator. Transfer 0.4121 g of the dried salt to a tared 250 mL volumetric flask, dilute to 250.0 g with reagent water and mix well. Store this primary standard in a 250 mL plastic container in a refrigerator; prepare a new primary standard annually.

10.4 Fluoride stock solution (100 mg F^-/L)

Purchase a certified 1000 ppm standard. Transfer 10.00 g of standard to tared 100 mL volumetric flask, dilute to 100.0 g with reagent water and mix well. Store certified standard in its own container and the working stock in a 125 mL plastic container in a refrigerator; prepare the working stock fresh every six months. Purchase a new certified standard annually.

10.5 Nitrogen (as NO_3^-) stock solution (100 mg N/L)

Purchase 1000 ppm certified standard. Transfer 10.00 g of standard to tared 100 mL volumetric flask, dilute to 100.0 g with reagent water and mix well. Store certified standard in its own container and the working stock in a 125 mL plastic container in a refrigerator; prepare the working stock fresh every month. Purchase a new certified standard every six months.

10.6 Nitrogen (as NO_2^-) stock solution (100 mg N/L)

Purchase 1000 ppm certified standard. Transfer 10.00 g of standard to tared 100 mL volumetric flask, dilute to 100.0 g with reagent water and mix well. Store certified standard in its own container and the working stock in a 125 mL plastic container in a refrigerator; prepare the working stock fresh every month. Purchase a new certified standard every six months.

10.7 Phosphate (as P) stock solution (100 mg P/L)

Purchase 1000 ppm certified standard. Transfer 10.00 g of standard to tared 100 mL volumetric flask, dilute to 100.0 g with reagent water and mix well. Store certified standard in its own container and the working stock in a 125 mL plastic container in a

refrigerator; prepare the working stock fresh every six months. Purchase a new certified standard annually.

10.8 Sulfate stock solution (1000 mg SO_4^{-2}/L)

Purchase a certified standard. Alternatively, dry approximately 0.6 g of potassium sulfate (K_2SO_4) in an oven for 30 minutes at 105°C, and cool in a desiccator. Transfer 0.4535 g of the dried salt to a tared 250 mL volumetric flask, dilute to 250.0 g with reagent water and mix well. Store this primary standard in a 250 mL plastic container in a refrigerator; prepare a new primary standard annually.

10.9 Working Calibration Standards (Prepare monthly unless nitrite, nitrate and phosphate are included in the standard, then the standards should be prepared weekly.)

Using deionized water as a zero standard, prepare six calibration standards from the working stock standards described above. Prepare Standard A (Level 6) by transferring 10.0 g each of the fluoride, chloride, nitrite-nitrogen, bromide, nitrate-nitrogen, and phosphate working standards and 20.0 g of the sulfate working standard to a tared 200 mL volumetric flask, dilute to 200.0 g with reagent water and mix well. Prepare Standard B (Level 5) by transferring 40.0 g of Standard A to a tared 100 mL volumetric flask, dilute to 100.0 g with reagent water and mix well. Prepare Standard C (Level 4) by transferring 20.0 g of Standard A to a tared 100 mL volumetric flask, dilute to 100.0 g with reagent water and mix well. Prepare Standard D (Level 3) by transferring 20.0 g of Standard A to a tared 200 mL volumetric flask, dilute to 200.0 g with reagent water and mix well. Prepare Standard E (Level 2) by transferring 5.0 g of Standard A to a tared 250 mL volumetric flask, dilute to 250.0 g with reagent water and mix well. Prepare Standard F (Level 1) by transferring 5.0 g of Standard A to a tared 500 mL volumetric flask, dilute to 500.0 g with reagent water and mix well.

NOTE: If only some of the above anions are of interest in the assay, the calibration standards may be prepared as a mixture of just those anions of interest. All seven of the anions do not have to be included in the standards unless the sample is to be analyzed for all seven. All standards are stored refrigerated.

11.0 Sample Handling

11.1 Collect 250 mL of sample in a container free of the analytes of interest, and if analysis cannot be performed immediately after collection, store samples in a refrigerator at ~ 4° C. All samples collected for nitrate, nitrite and/or orthophosphate analysis should be analyzed as soon as possible after collection and must be analyzed within 48 hours of collection without any chemical preservation.

12.0 Procedure

NOTE - Special training is required to operate the Lachat QuikChem 8000 Ion Chromatograph. Only those analysts who have received the required training will be able to operate the instrument and follow the procedure outlined below. The following procedure assumes that a valid method has already been created.

12.1 Preliminary Preparations for Analysis

12.1.1 Turn on the IC, autosampler, eluent pump, peristaltic pump and system computer modules.

12.1.2 Prepare sufficient quantities of fresh eluent and regenerant solutions.

12.1.3 Fill the probe rinse and deionized water reservoirs with reagent water.

12.1.4 Degas the eluent, regenerant and 4 liters of manifold deionized water using a high-purity helium sparger for one minute per liter of reagent.

12.1.5 Place the reagent lines in the appropriate reagent reservoir containing prepared reagent.

12.1.6 Set and clamp the pump tubes on the peristaltic pump.

12.1.7 Prime the eluent pump with eluent using the 30 mL syringe.

12.1.8 Enter the Instrument program which will automatically open Method "epa300a.met".

12.1.9 Preview the instrument baseline and verify stability for a ten-minute period of time.

12.1.10 Prepare a set of calibration standards.

12.1.11 Submit a mid-range standard to verify that column conditions and detector response appear normal.

12.2 Sample preparation and analysis

12.2.1 Create a batch of calibration standards, samples and the requisite QC samples, entering all tray assignments of the batch into the IC Prep Log.

12.2.2 Filter all samples using a new 0.45 µm membrane filter for each sample and QC sample.

12.2.3 Submit the batch for the analysis.

12.3 Instrument Shut-down

12.3.1 Place all manifold lines in deionized water and flush for 10 minutes.

12.3.2 Remove all manifold lines from the deionized water and flush with air for 5 minutes.

12.3.3 Place the eluent pump line in deionized water, prime, and flush columns for 10 minutes.

12.3.4 If instrument will not be used for more than 2 days, cap the columns and place in refrigerator at 4° C to inhibit bacterial growth.

12.3.5 Unclamp peristaltic pump tubing.

12.3.6 Exit Program from the system computer and turn off computer.

13.0 Data Analysis and Reporting

13.1 Review the calibration curves for each anion. The correlation coefficient for each curve must be equal to or greater than 0.997 using a cubic curve regression. If the correlation coefficient of one of the curves does not meet this criteria, the data from the curves that pass are still valid, but the entire batch must be reanalyzed for the one anion that did not pass.

NOTE: For the analysis of drinking water samples, the method must calculate the standard deviation of the retention time values for each parameter and set retention time windows at ± 3σ. Do not report the result from a peak falling outside of the 3σ window; investigate all potential sources of error or matrix effects and reanalyze.

13.2 Print a custom report which includes sample identification, date of analysis, time of analysis, analyst, method, run-time chromatogram, retention times, peak areas and calculated values for each anion.

13.3 Print the Batch report and calibration curves for each anion.

13.4 Indicate on the instrument report all valid results, exactly as they will be reported.

13.5 Prepare a final data report and submit for peer-review. Make any necessary corrections.

13.6 Enter only valid results into the LIMS system for final reporting. Include the appropriate condition code for any results that must be qualified (see Table 1).

13.7 Print the data entry table for data entry peer-review. Make any necessary corrections.

13.8 File the final data report.

14.0 QA/QC

14.1 A Lab Method Blank (LMB) must be analyzed at the beginning of every batch and every ten samples thereafter. If the result is not less than the reporting limit for the analyte(s) of interest (refer to PQLs listed in Reporting Limitations section), the analyst must reanalyze the blank to confirm the failure. If the failure is confirmed, the entire batch must be reanalyzed.

14.2 A duplicate analysis must be analyzed with every analytical batch at a frequency of every ten samples. Calculate the relative percent difference (RPD) as follows:

$$RPD = 100 (| X_1 - X_2 |) / m$$

where X_1 = first duplicate result
X_2 = second duplicate result
m = arithmetic average of X_1 and X_2

If the RPD is greater than the control limit charted by LIMS, the analyst must consult the Laboratory Supervisor for possible reanalysis and/or reporting of a matrix effect for that individual sample or the entire QC batch.

14.3 A matrix spike analysis must be analyzed with every analytical batch at a frequency of every ten samples. The spike concentration must be no more than five times and no less than one-fifth the native concentration of analyte. Calculate the matrix spike recovery as follows:

$$\% \text{ Recovery} = 100 (M - S) / a$$

where M = spiked sample result, mg/L
S = sample result, mg/L
a = matrix spike addition, mg/L

If a matrix spike recovery falls outside the control limit range charted by LIMS, the analyst must first confirm the failure by reanalyzing. If confirmed, consult the Laboratory Supervisor for possible reanalysis and/or reporting of a matrix effect for that individual sample or the entire QC batch.

14.4 A Calibration Verification Standard (CVS) must be analyzed at the beginning of every analytical batch and after every ten samples. The LCS must originate from a second standard source and represent a mid-range calibration check. Calculate the percent recovery as follows:

$$\% \text{ Recovery} = 100 \times C / t$$

where C = the control standard result, mg/L
t = the true value of the standard, mg/L

If the percent recovery falls outside of the 95% confidence limits certified by the manufacturer, the failure must be confirmed by reanalysis. If confirmed, the entire batch must be reanalyzed on a new calibration.

15.0 Reporting Limitations

16.1 Report only valid results based on the acceptance criteria outlined in the Data Analysis and QA/QC sections of this SOP. Report results to one decimal place and a maximum of three significant figures.

The Practical Quantitation Limit (PQL) for each analyte is listed below:

Parameter	PQL, mg/L
Fluoride	0.05
Chloride	0.5
Nitrite	0.05

Bromide	0.05
Nitrate	0.05
Phosphate	0.05
Sulfate	1.0

Signature Page

To the best of my knowledge all information contained in this document is complete and accurate.

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Dave Alkema

Supervisor: Michael Jaeger Date: 10/9/06
Michael Jaeger

Laboratory Director: Michael Jaeger Date: 10/9/06
Michael Jaeger

QA Approval: Linda Felcyn Date: Oct. 10, 2006
Linda Felcyn

STANDARD OPERATING PROCEDURE

Quantitation of Alkalinity in Water by Titration

1.0 References

1.1 *Standard Methods for the Examination of Water and Wastewater*, 18th edition, Method 2320 B and 4500-CO_2 D.

1.2 *Annual Book of ASTM Methods*, Volume 11.01, D 513 Method C.

1.3 *Measurement of pH in Water by the Electrometric Method*, KAR Laboratories, Inc., SOP KC420.

2.0 Application

2.1 This method is suitable for the determination of the various types of alkalinity in nearly all waters, aqueous extracts and aqueous filtrates.

3.0 Discussion

3.1 The alkalinity of a water is a measure of its capacity to react with a strong acid to a specific endpoint. The most common approach to measuring alkalinity in water assumes that hydroxide, carbonate and bicarbonate anions are the only species contributing to alkalinity.

3.2 Some specific terminology is used to describe the various types of alkalinity. Total alkalinity is the alkalinity of a sample titrated from its initial pH down to pH 4.5. Phenolphthalein alkalinity is the alkalinity of a sample titrated from its initial pH down to pH 8.3. Hydroxide alkalinity is the alkalinity of the sample titrated from its initial pH down to pH 8.3 but with the carbonate contribution subtracted by way of a second titration from pH 8.3 to 4.5. Determinations for carbonate and/or bicarbonate are considered analyses for alkalinity and are often referred to as "carbonate alkalinity" and "bicarbonate alkalinity."

3.3 Carbon dioxide species exist in water as three different forms **in equilibrium**: carbonate, bicarbonate and carbonic acid. The pH of the aqueous matrix determines the particular form which will predominate. Above pH 8.3 the carbonate anion ($CO_3^{=}$) predominates and from pH 8.3 to 4.5, the bicarbonate anion (HCO_3^{-}) predominates. A good understanding of what is occurring chemically during these titrations will assist the analyst in the final calculations.

3.4 When the initial pH of a sample is slightly above 8.3, the sample usually contains both carbonate and bicarbonate species. The sample is first titrated to pH 8.3. This converts nearly all of the carbonate to bicarbonate. The sample is then titrated to pH 4.5, converting the bicarbonate almost entirely to carbonic acid. It is important to note that the second titration to pH 4.5 converts both the bicarbonate that was originally present in the sample and the bicarbonate that was created by titrating the carbonate. This explains the purpose of the (B-A) term in some of the bicarbonate calculations; the bicarbonate created by titrating the carbonate must be subtracted in order to determine the concentration of bicarbonate that was in the sample originally.

3.5 Occasionally, a client will request a carbonate determination on a sample whose initial pH is less than 8.3. Even though the pH is in the "bicarbonate" region, some carbonate will be present but in a very small amount. The carbonate result is calculated by applying the equilibrium constant to the amount of bicarbonate present. This explains the exponential term in some of the carbonate calculations.

3.6 Occasionally, a bicarbonate analysis will be requested on a sample which contains hydroxide alkalinity. The sample will have a high initial pH and require more titrant to achieve pH 8.3 than that needed to go from pH 8.3 to pH 4.5. The analyst should then report a non-detect result for bicarbonate and report the hydroxide and/or carbonate alkalinity present.

3.7 In some cases, this titration method can be used to determine the alkalinity of a solid material (soil, scale, etc.) The method is most applicable to those materials that will dissolve readily in water or the hydrochloric acid titrant. With these samples, only alkalinity can be determined; not carbonate or bicarbonate (unless the material is known to be composed of just carbonate or bicarbonate as the anion). Prior to titrating, the solid material should be supported with a small quantity of water to give the material an environment for measuring pH and encouraging dissolution. Use the calculation for total alkalinity using the mass of sample in place of sample volume.

4.0 Summary of Method

4.1 The initial pH of a 50 mL aliquot of a sample is measured. The sample is then titrated with a standard hydrochloric acid titrant to pH 8.3 and then to pH 4.5. The volume of titrant required to achieve each of these pH values is recorded separately. The particular alkalinity being requested by the client is then calculated from these titrant volumes.

5.0 Sample Handling

5.1 A minimum of 250 mL of sample should be collected in a glass or plastic bottle. The sample must be analyzed within 14 days of collection. Refrigerate the sample until analysis.

6.0 Interferences

6.1 The presence of silt in monitoring well samples is a major concern for a positive interference. Do **not** filter a silty sample except in cases where the client has

instructed a sample to be filtered. If a sample contains suspended solids, mix the sample gently, pour off approximately 250 mL into a 250 mL plastic container and set the container in the refrigerator to allow the solids to settle overnight. Then pour off 100 mL from that 250 mL container to use for the analysis.

6.2 Do not stir the sample too quickly as stirring the sample during titration will encourage sample to pick up carbon dioxide causing a positive interference. Do not titrate the sample too slowly as any stirring of the sample will cause the sample's pH to drift slowly over time.

6.3 Be alert to all possible interferences for pH measurement. Soaps, oils, greases, suspended solids or precipitates may coat the pH electrode and cause a sluggish response. Cleaning the electrode between samples may be necessary. Allow additional time between titrant additions to allow the electrode to stabilize if junction becomes contaminated.

7.0 Apparatus

7.1 pH meter with standard combination electrode
Buret, 50 mL, with Teflon stopcock
Stir plate with 1" magnetic stir bars
Beakers, 100 mL, plastic, disposable
Graduated cylinder, 50 mL, Class A
Volumetric pipet or pipettor, 5.0 mL
Watchglass, glass, 4"

8.0 Chemicals and Reagents

8.1 Hydrochloric acid titrant
pH Buffers

9.0 Safety

9.1 Even dilute hydrochloric acid solutions are extremely corrosive to the eye. The same is true for sodium hydroxide solutions. Wear safety glasses and latex gloves when handling and titrating with a hydrochloric acid or sodium hydroxide solution.

10.0 Preparation of Standards

10.1 Hydrochloric Acid Titrant

Purchase a fresh, certified standard of 0.020 N and 0.050 N hydrochloric acid. Store at room temperature and observe manufacturer's expiration date.

10.2 pH Buffers

Purchase certified buffers with values of 4.0, 7.0 and 10.0 for calibrating the pH meter system. Store at room temperature and observe manufacturer's expiration date.

10.3 Sodium Hydroxide Check Standard, 0.10 N

Purchase a fresh certified standard. Store at room temperature and observe the manufacturer's expiration date.

11.0 Procedure

11.1 Calibrate the pH meter-electrode system using room-temperature buffers as outlined in KAR SOP KC420. Record the date, analyst initials, sample ID(s), the buffers used for calibrating, and the slope of the pH calibration.

11.2 Using a graduated cylinder, transfer 50.0 mL of sample to a 100 mL plastic disposable beaker. Add a clean stir bar, cover the beaker with a watchglass and allow the sample to come to room temperature. Record the amount of sample to be titrated.

NOTE: In samples where the alkalinity is expected to be less than 100 mg/L, the analyst should titrate 100 mL of sample. The beaker must be able to accommodate the volume of titrant added.

11.3 Select the titrant with a concentration that will most efficiently and accurately neutralize the amount of sample being titrated. Record the concentration and lot number of the titrant being used. For a 50 mL aliquot of most groundwater samples the 0.05 N titrant is preferred. A volume of 5–30 mL of titrant is ideal. If a sample requires less than 2.0 mL of the 0.05 N titrant, the analyst should set up a 100 mL aliquot of the sample and use the 0.02 N titrant.

11.4 Load the buret by rinsing the inside of it with a small portion of titrant and discarding the waste; then fill the buret with titrant. Ensure there is no air bubble in the buret tip below the stopcock.

11.5 Position the pH electrode holder, buret and stir plate so that the pH of a sample can be monitored continuously during titration.

11.6 Verify the accuracy of the titrant by pipetting 5.0 mL of the 0.10 N Sodium Hydroxide Check Standard (use a volumetric pipet or pipettor) into approximately 40 mL of deionized water. Add a stir bar and place the beaker on the stir plate with moderately slow stirring. Record the initial buret reading, immerse the electrode, and titrate the standard to pH 7.0. Record the final buret reading.

11.6.1 Calculate the normality of the titrant using the following equation:

$$\text{normality of titrant} = \frac{(V_{std} \times N_{std})}{V_t}$$

where V_t = mL of titrant (final buret reading minus initial buret reading)
N_{std} = normality of standard
V_{std} = mL of standard

11.6.2 The normality of the titrant must fall within 5 % of its certified value in order to use the certified value. If the titrant falls outside of the acceptable range, verify the titrant's concentration with a second titration. If it also falls outside the range, use the average of the two readings as the actual titrant concentration.

11.7 Set the sample prep on the stir plate, stir at a slow to moderately slow rate, and remove the watchglass. Immerse the pH electrode into the sample, allow the reading to stabilize, and record the initial sample pH.

11.8 If the sample pH is below 4.5 (or the endpoint required for the analysis), there is no need to titrate the sample. Record that the initial pH is lower than the endpoint, and see Section 14 for reporting.

11.9 Record the initial buret reading. Starting with a dropwise addition of titrant, titrate the sample to the pH 8.3 endpoint (if the sample pH is above 8.3). Try to catch the endpoint exactly. In some cases, a partial drop is needed near the endpoint. Record the buret reading.

NOTE: Do not repeat the analysis if the endpoint was exceeded due to the addition of a single drop. If adding more than a drop when near the endpoint caused an overshot, repeat the analysis.

11.10 Continue the titration to the pH 4.5 endpoint. Record the final buret reading along with any relevant comments or problems encountered during the titration.

12.0 Calculations

12.1 Using the various buret readings recorded during the titration, let

A = mL HCl titrant to pH 8.3
B = mL HCl titrant from pH 8.3 to 4.5
N = normality of HCl titrant

12.2 If the initial pH is less than or equal to 8.3, then:

Bicarbonate (as HCO_3^-) mg/L = $\frac{B \times N \times 61{,}000}{\text{mL sample}}$

(as $CaCO_3$) mg/L = $\frac{B \times N \times 50{,}000}{\text{mL sample}}$

Carbonate (as $CO_3^=$) mg/L = $\frac{B \times N \times 30{,}000}{\text{mL sample}} \times 0.94 \times 10^{(pH-10)}$

(as $CaCO_3$) mg/L = $\frac{B \times N \times 50{,}000}{\text{mL sample}} \times 0.94 \times 10^{(pH-10)}$

Hydroxide alkalinity is <5 mg/L (as $CaCO_3$) or <5 mg/L (as OH^-).
Phenolphthalein alkalinity is <5 mg/L (as $CaCO_3$).

12.3 If the initial pH is greater than 8.3, and A is less than B, then:

Bicarbonate (as HCO_3^-) mg/L = $\frac{(B-A) \times N \times 61,000}{mL\ sample}$

(as $CaCO_3$) mg/L = $\frac{(B-A) \times N \times 50,000}{mL\ sample}$

Carbonate (as $CO_3^=$) mg/L = $\frac{2A \times N \times 30,000}{mL\ sample}$

(as $CaCO_3$) mg/L = $\frac{2A \times N \times 50,000}{mL\ sample}$

Hydroxide alkalinity is <5 mg/L (as $CaCO_3$) or <5 mg/L (as OH^-).

12.4 If the initial pH is greater than 8.3, and B is less than A, then:

Carbonate (as $CO_3^=$) mg/L = $\frac{2B \times N \times 30,000}{mL\ sample}$

(as $CaCO_3$) mg/L = $\frac{2B \times N \times 50,000}{mL\ sample}$

Hydroxide (as OH^-) mg/L = $\frac{(A-B) \times N \times 17,000}{mL\ sample}$

(as $CaCO_3$) mg/L = $\frac{(A-B) \times N \times 50,000}{mL\ sample}$

Bicarbonate is <5 mg/L (as HCO_3^-) and <5 mg/L (as $CaCO_3$).

12.5 Regardless of the initial pH:

Total Alkalinity (as $CaCO_3$) mg/L = $\frac{(A+B) \times N \times 50,000}{mL\ sample}$

12.6 In samples where the alkalinity is due almost entirely to hydroxides, carbonates and bicarbonates and the total dissolved solids is less than 500 mg/L, the various forms of alkalinity, Free Carbon Dioxide and Total Carbon Dioxide can be calculated from a pH and total alkalinity analysis performed at 25˚C:

Bicarbonate, mg/L (as $CaCO_3$) = $\frac{T - \{5.0 \times 10^{(pH-10)}\}}{1 + \{0.94 \times 10^{(pH-10)}\}}$ T = Total Alk.

Carbonate, mg/L (as $CaCO_3$) = $0.94 \times B \times 10^{(pH-10)}$ B = Bicarbonate

Hydroxide, mg/L (as $CaCO_3$) = $5.0 \times 10^{(pH-10)}$

Free Carbon Dioxide, mg/L (as CO_2) = $2.0 \times B \times 10^{(6-pH)}$ B = Bicarbonate

Total Carbon Dioxide, mg/L (as CO_2) = $A + 0.44(2B + C)$

where A = Free Carbon Dioxide
B = Bicarbonate Alkalinity
C = Carbonate Alkalinity

NOTE: The department supervisor's PC has a calculating table which can perform these calculations very quickly. The file is My Documents/CO2.wpd.

13.0 QA/QC

13.1 Perform one blank analysis every analytical batch on 100 mL of deionized water. If the result is not non-detect, investigate all potential sources of error and correct any problems before reporting any sample results.

13.2 Perform one duplicate analysis for every ten samples. If the relative difference between the two duplicate results is greater than 10 %, the analyst should not report any results from that batch until all possible sources of error have been investigated.

13.3 Perform the titrant verification analysis described in section 11.7 once every analytical batch.

14.0 Reporting

14.1 Except for Carbonate, report all alkalinity results to the whole number with no more than 3 significant figures. Carbonate results should be reported to the 0.01 unit with no more than 3 significant figures.

14.2 The limit of detection for this method is 5 mg/L for total, phenolphthalein, hydroxide, and bicarbonate alkalinity; 0.02 mg/L for carbonate alkalinity. Unless the client requests otherwise, carbonate results should be reported (as CO3), bicarbonate results (as HCO3) and hydroxide alkalinity (as OH). Phenolphthalein and total alkalinity results are always reported (as CaCO3).

14.3 In those cases where the initial pH of the sample is already below the pH 4.5 endpoint, report a result of *See comment*, remove the default units, attach the AJ code, and enter XXX for the analyst initials so that the client will not be charged for work that was not performed.

Signature Page

To the best of my knowledge all information contained in this document is complete and accurate.

Author:_____ Date:_____
Dave Alkema

Supervisor:_____ Date:_____
Michael Jaeger

Laboratory Director:_____ Date:_____
Michael Jaeger

QA Approval:_____ Date:_____
Pollyann Nee

QAPP Worksheet #37 Usability Assessment

- retrieval of missing information
- request for additional explanation or clarification
- reanalysis of sample from extract (when appropriate)
- recalculation or reinterpretation of results by the laboratory

These actions may improve the data quality, reduce uncertainty and eliminate the need to qualify or reject data.

If these actions do not improve the data quality to an acceptable level, the following additional actions may be taken:

- extrapolation of missing data from existing data points
- use of historical data
- evaluation of the critical/noncritical nature of the sample

If the data gap cannot be resolved by these actions, the data bias and potential for false negatives and positives can be evaluated. If the resultant uncertainty level is unacceptable, the following action must be taken:

- additional sample collection and analysis