URS

323743

July 20, 2007

Ms. Terese Van Donsel
United States Environmental Protection Agency
Office of Superfund, Region 5
SR-6J
77 West Jackson
Chicago, IL 60604-3590

Re: Submittal of Revised QAPP
Detrex Corporation
Detrex Source Control Operable Unit -- Fields Brook Superfund Site
Docket No. V-W-98-C-450

Dear Ms. Van Donsel:

On behalf of Detrex Corporation, URS Corporation is submitting the Revised Quality Assurance Project Plan (QAPP) based on the EPA comments.

If you have any questions regarding this submittal, please do not hesitate to contact either Tom Steib at (440) 397-6131 or me at (216) 622-2432.

Sincerely,

URS Corporation

Martin L. Schmidt

Martin L. Schmidt, Ph. D.
Project Manager

MLS/mg

Attachment

cc: Pete Felitti – USEPA
Sig Williams – OEPA
Tom Steib - Detrex
Tom Doll – Detrex
Bob Currie – Detrex

URS Corporation
1375 Euclid Ave., Suite 600
Cleveland, OH 44115-1808
Tel: 216.622.2400
Fax: 216.622.2428

P:\D\Detrex\13811443\DOCs\Correspond\Letters\VanDonsl_Comment-Response_072007.doc

Detrex Corporation Comment Response Report to
U.S. EPA Comments on URS June 8, 2007 Responses to the Detrex Source Control Area RD/RA QAPP
Quality Assurance Project Plan (QAPP)
Detrex Source Control Operable Unit – Fields Brook Superfund Site
Ashtabula, Ohio
Docket No. V-W-98-C-450

This Comment Response Report is in response to the U.S. Environmental Protection Agency's (U.S. EPA) Comments on the URS Responses to the Detrex Source Control Area RD/RA QAPP (February 2007 *Draft Quality Assurance Project Plan (QAPP) Remedial Design/Remedial Action (RD/RA)* dated May 15, 2007). Detrex Corporation (Detrex) is responding as follows:

U.S. EPA Comment No. 1 (QAPP Worksheet #10):
(1) The following **URS** response to QAPP Worksheet #10 needs to be addressed.

> U.S. EPA Comment No. 5 (QAPP Worksheet #10 Page 17 of 88, 2nd bullet):
>
> There is a double negative statement in the second sentence. Please clarify.
>
> Detrex Corporation Comment Response No. 5 (QAPP Worksheet #10 Page 17 of 88, 2nd bullet):
>
> Detrex and URS have not been able to verify this comment.

QAPP Worksheet #10 with the double negative statement encircled was faxed to URS on June 15, 2007. Please clarify the statement.

Detrex Corporation Comment Response No. 1 (QAPP Worksheet #10):

The word "no" has been removed from the second sentence eliminating the double negative.

(2) The following **URS** Response to the second comment on QAPP Worksheet #15 concerning PCB analysis in Water/Surface Water was not addressed.

> Verify if PCBs should be included for the Matrix Water analysis. QAPP Worksheets
> #12, #15, and #23 imply PCB analysis in Water/Surface Water.

Detrex Corporation Comment Response No. 2 (QAPP Worksheet #15):

PCBs are not being sampled in the water matrix at this time. However, at the request of the US EPA Region 5 RPM in our initial scoping meeting and in line with the current EPA guidance documents, PCBs have been included on worksheet #15 in the event that future work at the site dictates analysis of this parameter.

The **URS** proposed revisions have been incorporated into the First Revision of the QAPP which is attached. The QAPP revisions and the **FIRSTECHNOLOGY, Inc.** revisions to the laboratory standard operating procedures will sent under separate cover.

Workbook for
Uniform Federal Policy for Quality Assurance Project Plan

For the
Detrex Source Control Area
Remedial Design/Remedial Action
Operation, Maintenance and DNAPL Recovery System
Ashtabula, Ohio

Prepared by the URS Corporation
Cleveland, Ohio

For the Detrex Corporation
Cleveland, Ohio

Draft
Version 2
July 2007

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TABLE OF CONTENTS

Introduction 1
QAPP Worksheet #1. Title and Approval Page 2
QAPP Worksheet #2. QAPP Identifying Information 3
QAPP Worksheet #3. Distribution List 8
QAPP Worksheet #4. Project Personnel Sign-Off Sheet 9
QAPP Worksheet #5. Project Organizational Chart 10
QAPP Worksheet #6. Communication Pathways 11
QAPP Worksheet #7. Personnel Responsibilities and Qualification Table 12
QAPP Worksheet #8. Special Personnel Training Requirements Table 13
QAPP Worksheet #9. Project Scoping Session Participants Sheet 14
QAPP Worksheet #10. Problem Definition 15
QAPP Worksheet #11. Project Quality Objectives/Systematic Planning Process Statements 16
QAPP Worksheet #12. Measurement Performance Criteria Table 18
QAPP Worksheet #13. Secondary Data Criteria and Limitations Table 26
QAPP Worksheet #14. Summary of Project Tasks 27
QAPP Worksheet #15. Reference Limits and Evaluation Table 28
QAPP Worksheet #16. Project Schedule Timeline Table 52
QAPP Worksheet #17. Sampling Design and Rationale 53
QAPP Worksheet #18. Sampling Locations and Methods/SOP Requirements Table 54
QAPP Worksheet #19. Analytical SOP Requirements Table 55
QAPP Worksheet #20. Field Quality Control Sample Summary Table 56
QAPP Worksheet #21. Project Sampling SOP References Table 57
QAPP Worksheet #22. Field Equipment Calibration, Maintenance, Testing, and Inspection Table 58
QAPP Worksheet #23. Analytical SOP References Table 59
QAPP Worksheet #24. Analytical Instrument Calibration Table 60
QAPP Worksheet #25. Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table 61
QAPP Worksheet #26. Sample Handling System 62
QAPP Worksheet #27. Sample Custody Requirements 63
QAPP Worksheet #28. QC Samples Table 64
QAPP Worksheet #29. Project Documents and Records Table 65
QAPP Worksheet #30. Analytical Services Table 66
QAPP Worksheet #31. Planned Project Assessments Table 67
QAPP Worksheet #32. Assessment Findings and Corrective Action Responses 68
QAPP Worksheet #33. QA Management Reports Table 69
QAPP Worksheet #34. Verification (Step I) Process Table 70
QAPP Worksheet #35. Validation (Steps IIa and IIb) Process Table 71
QAPP Worksheet #36. Validation (Steps IIa and IIb) Summary Table 72
QAPP Worksheet #37. Usability Assessment 73

WORKBOOK FOR UNIFORM FEDERAL POLICY FOR QUALITY ASSURANCE PROJECT PLAN FOR THE DETREX SOURCE CONTROL AREA REMEDIAL DESIGN/REMEDIAL ACTION OPERATION, MAINTENANCE AND DNAPL RECOVERY SYSTEM ASHABULA, OHIO

INTRODUCTION

This *Workbook for Uniform Federal Policy for Quality Assurance Project Plan for the Detrex Source Control Area Remedial Design/Remedial Action Operation, Maintenance and DNAPL Recovery System Ashtabula, Ohio* provides the worksheets to assist in the preparation of the Quality Assurance Project Plan (QAPP) for this project in accordance with the UFP-QAPP (the UFP-QAPP Manual) and Section 6 (Part B) of *Quality Systems for Environmental Data and Technology Programs - Requirements with guidance for use*, ANSI/ASQ E4 (February 2004). This Workbook is being used by URS Corporation for the Detrex Corporation to assist with the preparation of the QAPP for environmental data gathering activities.

Each worksheet addresses specific requirements of the UFP-QAPP. Since the content and level of detail in a specific QAPP for the Detrex site some specific worksheets may not be applicable to this project.

The ultimate success of this project depends on the quality of the environmental data collected and data generated will be used in decision-making. The use of the worksheets will aid in identifying the critical project information that will ensure that the right type, quality, and quantity of data are collected to meet all of the project's quality objectives. Though the format of each worksheet is not mandatory, the information required on the worksheets must still be presented in the QAPP, as appropriate to the project.

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

Site Name/Project Name: Detrex Source Control Area
Site Location: Fields Brook Superfund Site - Ashtabula, Ohio

Document Title: Detrex RD/RA Quality Assurance Project Plan: DNAPL Recovery and Groundwater Monitoring at the Fieldbrook Superfund Site in Ashtabula, Ohio

Lead Organization: Detrex Corporation

Preparer's Name and Organizational Affiliation: David Blasuhild, URS Corporation

Preparer's Address, Telephone Number, and E-mail Address: 501 Holiday Drive, Suite 300, Pittsburgh, PA 15220, 412-503-4679, david blaushild@urscorp.com

Preparation Date (Day/Month/Year): July 20, 2007 – Revised Document

Investigative Organization's Project Manager/Date: _____
Signature
Printed Name/Organization: Martin Schmidt/URS Corporation

Investigative Organization's Project QA Officer/Date: _____
Signature
Printed Name/Organization: Peggy Schuler/URS Corporation and Peggy Schuler

Lead Organization's Project Manager/Date: _____
Signature
Printed Name/Organization: Thomas Steib/Detrex Corporation

Laboratory Director, FIRSTECHNOLOGY, Inc./Date: _____
Signature
Printed Name/Organization: Mark Kalmeyer/FIRSTECHNOLOGY, Inc.

Approval Signatures/Date: _____
Signature
Printed Name/Title: Terese A. Van Donsel

Approval Authority: Region 5, United States Environmental Protection Agency

Approval Signatures/Date: _____
Signature
Printed Name/Title: Region 5, United States Environmental Protection Agency, Quality Assurance Reviewer

Approval Authority: Region 5, United States Environmental Protection Agency

Other Approval Signatures/Date:_____
Signature

Printed Name/Title: Ohio EPA

Document Control Numbering System : _____

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

Site Name/Project Name: Detrex Source Control Area
Site Location: Fields Brook Superfund Site - Ashtabula, Ohio
Site Number/Code: V-W-98-C-450
Operable Unit:
Contractor Name: URS Corp.
Contractor Number:

Contract Title:
Work Assignment Number:

Title: Quality Assurance Project Plan
Revision Number: 01
Revision Date: February 23, 2--7
Page 3 of 43

1. Identify regulatory program: CERCLA
2. Identify approval entity: US Environmental Protection Agency Region 5
3. The QAPP is (select one): ☐Generic ☒Project Specific
4. List dates of scoping sessions that were held: January 10, 2007
5. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date
N/A	

6. List organizational partners (stakeholders) and connection with lead organization:
   Detrex Corporation, URS Corp., US EPA Region 5 (lead organization), State of Ohio, City of Ashtabula
7. List data users:
   US EPA Region 5; Detrex Corporation; State of Ohio, City of Ashtabula, URS Corp.
8. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusions below:

QAPP Worksheet #2
QAPP Identifying Information
(continued)

Identify where each required QAPP element is located in the QAPP (provide section, worksheet, table, or figure number) or other project planning documents (provide complete document title, date, section number, page numbers, and location of the information in the document). Type "NA" for the QAPP elements that are not applicable to the project. Provide an explanation in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	1
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	2
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	3 4
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	5 6 7 8
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)	9 10
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 PQOs - Measurement Performance Criteria Table	11/Laboratory QAPP 12

QAPP Worksheet #2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	NA NA
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	14 15 16
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 3.1.2.3 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.4 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	17 **Add Map** 18 19 20 21 22
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	23 24 25

QAPP Worksheet #2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Required Documents
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 Form and Seal	26 27 Laboratory QAPP Laboratory QAPP
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	28
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	29 30
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	31 NA
4.2 QA Management Reports	- QA Management Reports Table	33
4.3 Final Project Report		

QAPP Worksheet #2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related 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	34 35 36 37
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		NA

QAPP Worksheet #3

(UFP-QAPP Manual Section 2.3.1)

List those entities to whom copies of the approved QAPP, subsequent QAPP revisions, addenda, and amendments will be sent.

☐ Worksheet Not Applicable (State Reason)

Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Martin L. Schmidt	Project Manager	URS Corp.	216.622.2400	216.622.2464	martin schmidt@urscorp.com	
Peggy Schuler	QA Officer	URS Corp.	216.622.2400	216.622.2464	peggy schuler@urscorp.com	
David Blaushild	Project Chemist	URS Corp.	412.503.4679	412.503.4701	david blaushild@urscorp.com	
Tom Steib	Plant Manager	Detrex Corp.				
Tom Doll	Operations Manager	Detrex Corp.				
Terese VanDonsel	U.S. EPA PM	U.S. EPA	312.323.6564			
Richard Byvik	Quality Assurance Chemist	URS EPA Region 5	312-353-6564		Byvik.Richard@epamail.epa.gov	Detrex-03
Phil Byron	Manager of Business	Firstechnology Inc.	216.663.0808	216.663.0656	pbyron@firstechnology.com	
Scott Bolam	Quality Assurance Manger	Firstechnology Inc.	216-663-0808	216-663-0656	sbolam@firstechnology.com	Detrex-07
TBA	Field Team Leader	URS Corp.	216-622-2400	216-622-2464		Detrex-06

QAPP Worksheet #4 (UFP-QAPP Manual Section 2.3.2)
Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable sections of the QAPP and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

☐ Worksheet Not Applicable (State Reason)

Project Personnel Sign-Off Sheet

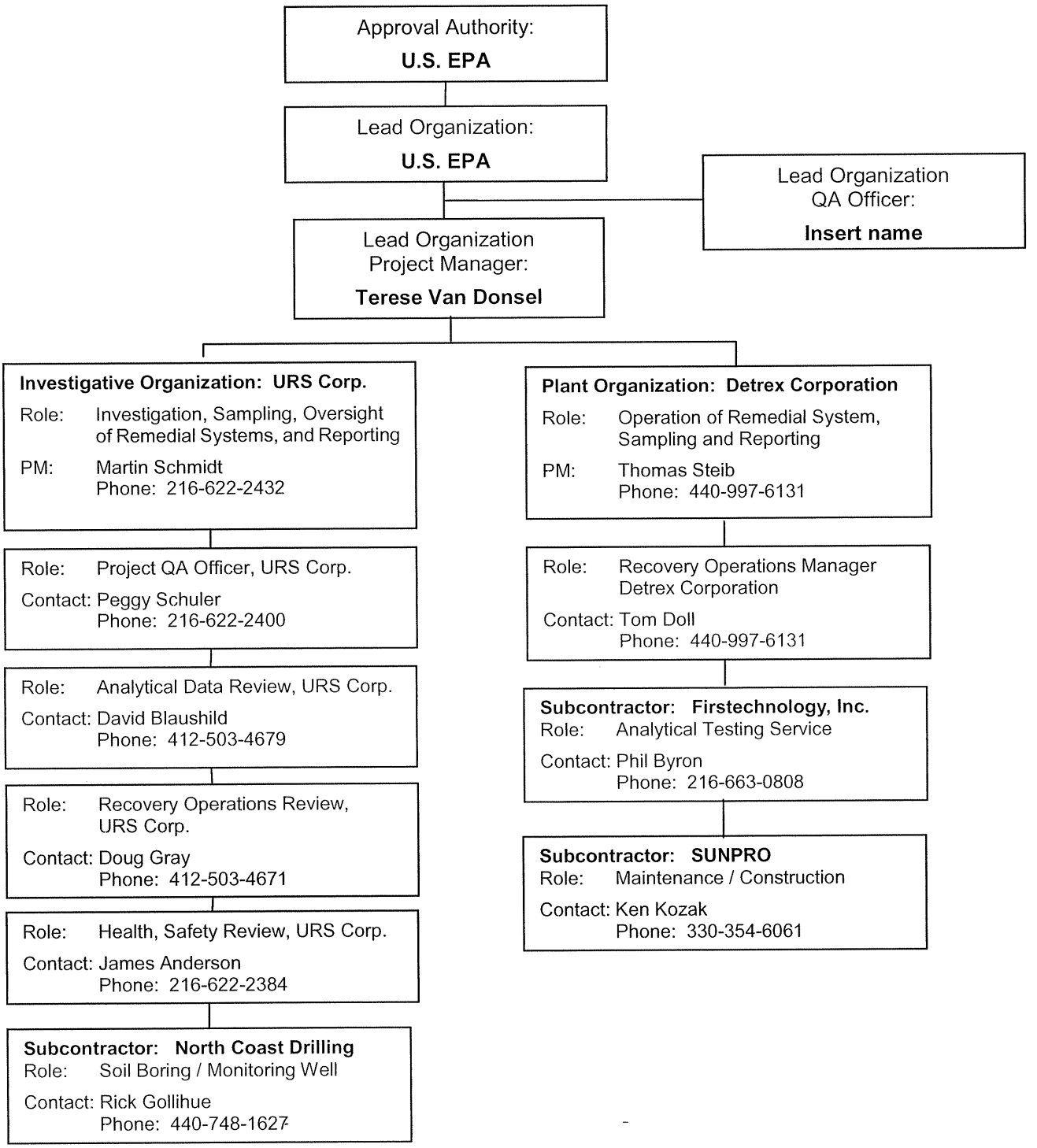
Organization: Detrex Corporation

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Terese A. Van Donsel	US EPA Regional Project Manager	312-353-6564		
Richard Byvik	US EPA Quality Assurance Officer	312-353-6564		
Thomas Steib	Project Manager, Detrex Corporation			
Tom Doll	Operations Manager, Detrex			
Martin Schmidt	Project Manager	216 622-2400		
Peggy Schuler	Quality Assurance Officer	216 622-2400		
Scott Bolam	Laboratory Quality Assurance Manager	216-663-0808		

QAPP Worksheet #5
(UFP-QAPP Manual Section 2.4.1)
Identify reporting relationships between all organizations involved in the project, including the lead organization and all contractor and subcontractor organizations. Identify the organizations providing field sampling, on-site and off-site analysis, and data review services, including the names and telephone numbers of all project managers, project team members, and/or project contacts for each organization.

☐ Worksheet Not Applicable (State Reason)

Project Organizational Chart



QAPP Worksheet #6

(UFP-QAPP Manual Section 2.4.2)

Describe the communication pathways and modes of communication that will be used during the project, after the QAPP has been approved. Describe the procedures for soliciting and/or obtaining approval between project personnel, between different contractors, and between samplers and laboratory staff. Describe the procedure that will be followed when any project activity originally documented in an approved QAPP requires real-time modifications to achieve project goals or a QAPP amendment is required. Describe the procedures for stopping work and identify who is responsible.

☐ Worksheet Not Applicable (State Reason)

Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Point of Contact with EPA RPM	Lead Organization Program Manager	Thomas Steib		All materials and information about the project with be forwarded to Teresa Van Donsel, EPA RPM
Manage all project phases	Contractor Project Manager	Martin Schmidt	216-622-2400	Martin Schmidt Ph.D. will the URS liason to Tom Steib. In addition, Dr. Schmidt will notify EPA and Mr. Steib of any field related problem by phone, fax, e-mail by the next business day.
QAPP Changes in the field	Sampling Team Leader			Field team leader will e-mail, phone in, or fax daily field reports to Dr. Schmidt.
Reporting Laboratory Data Quality Issues	Laboratory Quality Assurance Officer	Scott Bolan	216-633-0808	All QA/QC issues with project field samples will be reported to Peggy Schuler within 2 business days.
Field and Analytical Corrective Actions. Release of Analytical data.	Quality Assurance Officer	Peggy Schuler	216-622-2400	QA Officer Peggy Schuler will determine the need for corrective action for field and analytical issues. No analytical data can be released pior to validation and has been approved for release by Ms. Schuler or her designate.
QAPP Amendments	EPA Region 5 RPM	Terese Van Donsel	312-353-6564	Any major changes to the QAPP must be approved by Terese Van Donsel before changes can be implemented.

QAPP Worksheet #7

(UFP-QAPP Manual Section 2.4.3)

Identify project personnel associated with each organization, contractor, and subcontractor participating in responsible roles. Include data users, decision-makers, project managers, QA officers, project contacts for organizations involved in the project, project health and safety officers, geotechnical engineers and hydrogeologists, field operation personnel, analytical services, and data reviewers. Identify project team members with an asterisk (*). Attach resume to this worksheet or note the location of the resumes.

☐ Worksheet Not Applicable (State Reason)

Personnel Responsibilities and Qualification Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Martin Schmidt	Project Manager	URS Corporation	Project Oversight	Ph.D., 25 years exp.
David Blaushild	Project Chemist	URS Corporation	Data Review	20 years
Peggy Schuler	Project QA Officer	URS Corporation	Data Review	BS, 20 years exp.
Jeff Berk	Field Geologist/Mgr	URS Corporation	Coordination of Sampling	MS, 20 years exp.
Taki Shinkawa	Geologist	URS Corporation	Soil / GW Sampling	MS, 10 years exp.
Jerry Miller	Geologist	URS Corporation	Soil / GW Sampling	BS, 10 years exp.
James Anderson	Geologist	URS Corporation	HSSE Manager	BS, 15 years exp.
Doug Gray	Engineer	URS Corporation	Remedial Operations/Design	BS, 23 years exp.
Fred Coll	Geologist	URS Corporation	Remedial Data Review	MS, 13 years exp.
Tom Steib	Plant Manager	Detrex Corporation	Plan Operations/Reporting	
Tom Doll	Operations Manager	Detrex Corporation	Recovery Well Operations	
Phil Byron	Manager of Business	Firstechnology, Inc.	Laboratory	
Scott Bolam	Laboratory Quality Assurance Officre	Firstechnology, Inc.	Laboratory	

QAPP Worksheet #8

(UFP-QAPP Manual Section 2.4.4)

Provide the following information for those projects requiring personnel with specialized training. Attach training records and/or certificates to the QAPP or note their location.

☑ Worksheet Not Applicable (State Reason) not applicable to this project

Special Personnel Training Requirements Table

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Field Personnel	HAZWOPER (29cfr1910.120	urs	see HASP	see HASP	see HASP	see HASP

QAPP Worksheet #9

(UFP-QAPP Manual Section 2.5.1)

Complete this worksheet for each project scoping session held. Identify project team members who are responsible for planning the project.

☐ Worksheet Not Applicable (State Reason)

Project Scoping Session Participants Sheet

Project Name: Detrex **Projected Date(s) of Sampling:** **Project Manager:** Martin Schmidt	**Site Name:** Detrex Source Control Area **Site Location:** Fields Brook Superfund Site - Ashtabula, Ohio

Date of Session: January 10, 2007

Scoping Session Purpose: The purpose of the scoping session was to discuss project specific requirements for sampling and analysis at the site and the discuss QAPP format requirements.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Terese A. Van Donsel	Remedial Project Manager	US EPA Region 5	312-353-6564	Vandonsel.Terese @epamail.epa.gov	Remedial Project Managers
Richard Byvik	Quality Assurance Chemist	URS EPA Region 5	312-353-6564	Byvik.Richard@epamail.epa.gov	EPA QA Chemist
Martin Schmidt	Project Manager	URS Corp.	216-622-2400	martin schmidt@urscorp.com	Project Manager
David Blaushild	Project Chemist	URS Corp.	412-503-4679	david blaushild@urscorp.com	Quality Assurance Officer

Comments/Decisions: Determined the suite of parameters to be tested including VOCs, SVOCs, PCBs, and target analyte list (TAL) metals in sediment.. Laboratory will provide a level 4 data package (CLP-like). QAPP elements will be developed for soil, groundwater, surface water, sediment.

Action Items: Plan groundwater sampling events; select analytical methods, project quantitation limits and data quality objectives.

Consensus Decisions: Draft QAPP to be submitted to EPA Region 5 on or about February 23, 2007.

QAPP Worksheet #10

(UFP-QAPP Manual Section 2.5.2)

Clearly define the problem and the environmental questions that should be answered for the current investigation and develop the project decision "If..., then..." statements in the QAPP, linking data results with possible actions. The prompts below are meant to help the project team define the problem. They are not comprehensive.

☐ Worksheet Not Applicable (State Reason)

Problem Definition

The problem to be addressed by the project:

There are three components to this project that involve additional data collection activities that are covered in this QAPP. The overall project involves monitoring the effectiveness of an existing DNAPL Recovery System. Groundwater sampling onsite from monitoring wells and a down gradient groundwater collection trench and DNAPL monitoring in the existing source area. The following summaries are the key data collection activities.

1. *Groundwater monitoring – The remedy that has been installed at the Detrex facility includes a slurry wall, two groundwater collection trenches and active DNAPL removal using extraction wells. As part of the long-term performance and monitoring of site groundwater conditions, U.S. EPA has requested groundwater sampling of down gradient monitoring wells and the Southern Area down gradient groundwater collection trench. Sampling is anticipated to occur on a semi-annual basis from monitoring wells and as needed for the collection trench. The actual number of wells to be sampled has not been specified by U.S. EPA or Detrex at this time.*
2. *DNAPL Recovery and Monitoring – As described above, an active DNAPL Recovery System has been installed. As part of the recovery program, 2 additional recovery wells with a new design for well screen, sand pack, and pumps are planned for Spring 2007. Monitoring for the DNAPL Recovery System involves collection of total fluids and DNAPL. Recovery amounts are reported to U.S. EPA.*
3. *Soil and Sediment Sampling – In addition to the groundwater monitoring program for selected monitoring wells and the groundwater collection trench, selected soil and sediment sampling will occasionally occur. The subsurface soil sampling will occur both on site and off site to delineate subsurface conditions. Sediment sampling will occur in the DS Tributary, which is in an offsite area of the project site. The sampling data will be used to develop limits of excavation and determine the volume of sediment to be excavated in the DS Tributary.*

The environmental questions being asked:

There are several environmental questions that are being asked for the Long-Term Monitoring Program and DNAPL Recovery Program at the Detrex Facility. The following questions relate to this project site:

1. *Has DNAPL migrated to the Fields Brook stream channel from the Detrex Source Area?*
2. *Has DNAPL migrated to the DS Tributary channel from the Detrex Source Area?*
3. *Are there impacts from DNAPL to onsite groundwater? Note: the onsite groundwater has been determined to be a non usable water-bearing zone. There are no drinking water wells in the area. All drinking water is supplied by Municipal sources.*
4. *Are there impacts from DNAPL to offsite stream channel sediment in either Fields Brook or the DS Tributary?*

Observations from any site reconnaissance reports:
The Detrex site has been studied during RI phase of work in the early 1990s. In addition, sampling occurred onsite in 1997-1999 prior to construction of the slurry wall and groundwater remedy. This data has been reported to the U.S. EPA.

A synopsis of secondary data or information from site reports:
In June 2005, there was concern that DNAPL material had migrated from the Detrex source area and re-contaminated the sediment within the Fields Brook stream channel. During the past two years, several phases of investigation have been conducted on the Detrex property and in offsite locations on adjacent properties. Several pathways were hypothesized as pathways for migration. These possible pathways included subsurface soil channels and sand lenes, an abandoned closed storm sewer line west of the facility, an abandoned outfall and storm water pond.

Soil samples and groundwater samples have been collected at more than 100 locations onsite by Detrex Corporation to delineate subsurface conditions and evaluate site pathways. Soil and groundwater samples in the southern portion of the facility did not detect DNAPL or impacted groundwater in upland areas or adjacent to the former storm sewer. In order to provide long-term protection of Fields Brook, Detrex Corporation installed a groundwater interceptor trench between the source area and the Fields Brook stream channel. During construction no VOCs were detected in soil or groundwater. In January 2007, U.S. EPA indicted that observed DNAPL and VOCs/SVOCs in the Fields Brook stream channel was historical in nature and not related to ongoing DNAPL movement from Detrex source areas. As described in Item 1, groundwater samples will be collected from the recovery trench and groundwater monitoring wells to monitor performance of the remedy and site conditions.

Soil and groundwater sampling west of the site and existing slurry wall did detect VOCs and SVOCs. As a result, this area will need to be further sampled, to delineate groundwater impacts and establish down gradient monitoring locations.

Also, historical observations have been made, which indicate a sheen is occasionally observed near the abandoned storm water outfall at Fields Brook and in the DS Tributary. Subsurface soil sampling along the storm sewer pipe indicates this area is not an ongoing source of DNAPL. The sheen is possibly related to an area of sediment that was not previously remediated. This area is planned for additional sampling and remediation in 2007. The observations in the DS Tributary indicate additional sampling of sediment is required, prior to implementation of remedy.

The possible classes of contaminants and the affected matrices:
Based on the sampling that has occurred at this facility since the RI/FS in 1993, the possible classes of contaminants are Volatile Organic Compounds and Semi-Volatile Organic Compounds. The facility was historically involved in production of materials having these compounds. The matrices that have been impacted by Volatile Organic Compounds and Semi-Volatile Organic Compounds include: groundwater, surface water, subsurface soil, and sediment.

The rationale for inclusion of chemical and nonchemical analyses:
Since historical production activities at the facility involved production of product material that contains volatile organic and semi-volatile organic compounds, the rationale for inclusion of these compounds is based the nature these compounds and possible degradation compounds.

Information concerning various environmental indicators:
At this time, there is no available information regarding environmental indicators.

Project decision conditions ("If..., then..." statements):

The following project decision conditions have been developed for reviewing sampling results from groundwater, subsurface soil and sediment data:

- *If VOCs are detected in groundwater monitoring well samples then the data will be reviewed and verified. If VOCs are not detected in down gradient wells or in the interceptor trench groundwater then no action is needed. Sampling and operation of the recovery trench will be terminated and limited. If VOCs are detected in down gradient groundwater onsite in the Southern Area, then an evaluation of remedial alternatives will be considered.*
- *If DNAPL is observed in southern groundwater collection trench then the trench will continue to be pumped. If DNAPL or VOCs are not detected then the trench will not be pumped and monitored only.*
- *If DNAPL is observed in existing or newly installed recovery wells, then the wells will be operated and monitored for DNAPL recovery.*
- *If VOCs or SVOCs are detected in sediment or subsurface soil above the Fields Brook Sediment Operable Unit Clean Up Goals, then the data will be reviewed and an appropriate feasible remedial alternative will be developed and implemented that is consistent with current SOU clean up programs..*

QAPP Worksheet #11

(UFP-QAPP Manual Section 2.6.1)

Use this worksheet to develop project quality objectives (PQOs) in terms of type, quantity, andquality of data determined using a systematic planning process. Provide a detailed discussion of PQOs in the QAPP. List PQOs in the form of qualitative and quantitative statements. These statements should answer questions such as those listed below. These questions are examples only, however; they are neither inclusive nor appropriate for all projects.

☐ Worksheet Not Applicable (State Reason)

Project Quality Objectives /Systematic Planning Process Statements

Who will use the data? Data will be used by EPA Region 5 to predict the affect of VOCs and SVOCs on groundwater and VOCs, SVOCs, PCBs, and metals in the Fields Brook sediments at the site. All data gathered during the project will be archived for one year and stored on CD.
What will the data be used for? URS Corp. and Detrex will use the data to determine the extent of impacts to soil, groundwater, surface water and sediment at the site. The data will be used to gauge the effectiveness of the treatment system at the site.
What type of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques) Representative samples will be collected in the Detrex Sourse Area at the site. Sample locations and will be addressed in the Sampling and Analysis plan. Standard protocols for sample collection, handling and preparation and analytical methods will be used including EPA Method 5035 for the collection of VOCs in soils.
How "good" do the data need to be in order to support the environmental decision? Data will need to meet the criteria of validity and usability as determined by US EPA data validation guidelines for organics and inorganics.
How much data are needed? (number of samples for each analytical group, matrix, and concentration) Sampling personnel will collect quarterly groundwater samples from 16 monitoring wells at the site for VOCs, and SVOCs. The standard concentrations as spelled out in the individual SW-846 methods will be used to evaluate these data. Sediment samples will be collected for VOCs, SVOCs, PCBs, and metals to determine impact of contaminants at the site. Numbers of soil, sediments and surface water will be determined as conditions dictate.
Where, when, and how should the data be collected/generated? The data will be collected at the Detrex source area from existing monitoring wells.
Who will collect and generate the data? Field personnel from URS Corp. will collect the samples in accordance with accepted procedures and deliver these samples to the laboratory for analysis. The laboratory will generate the data using accepted EPA methods.
How will the data be reported? The data will be reported by the laboratory as a standard Level 4 (CLP-like) report on a CD in pdf format and as a excel® table. The data will be reviewed and reported in tabular format. Electronic files of these data are available if requested by EPA.
How will the data be archived? The data will be archived on CD and stored for a period of three years.

QAPP Worksheet #12

(UFP-QAPP Manual Section 2.6.2)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the data quality indicators (DQIs), measurement performance criteria (MPC), and QC sample and/or activity used to assess the measurement performance for both the sampling and analytical measurement systems. Use additional worksheets if necessary. If MPC for a specific DQI vary within an analytical parameter, i.e., MPC are analyte-specific, then provide analyte-specific MPC on an additional worksheet.

☐ Worksheet Not Applicable (State Reason)

Measurement Performance Criteria Table

Matrix	Groundwater/Surface Water
Analytical Group	VOA
Concentration Level	Low

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)
URS Sampling Procedure	EPA Method 8260B/SOP-OA-1000-VOCs(see Worksheet #23 for corresponding lab SOPs)	Precision	RPD=30	Field Duplicates	A
		Accuracy, Bias, Contamination	No target analytes above RL	Field Blank, Trip Blank, Laboratory Method Blank	S & A
		Accuracy/Bias	+/- 30	LCS	A
		Accuracy/Bias	surrogate recoveries meet laboratory specified criteria	Surrogate Spike (organics)	A
		Sensitivity	Method specific criteria for calibration factors	Low Point Calibration Curve	A
		Completeness	90%	Completeness Check	A

Measurement Performance Criteria Table

Matrix	Soil/Sediment
Analytical Group	VOA
Concentration Level	Low

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)
URS Sampling Procedure	EPA Methods 5035/8260B SOP-OA-1000-VOCs(see Worksheet #23 for corresponding lab SOPs)/	Precision	RPD=50	Field Duplicates	A
		Accuracy, Bias, Contamination	No target analytes above RL	Field Blank, Trip Blank, Laboratory Method Blank	S & A
		Accuracy/Bias	+/- 30	LCS	A
		Accuracy/Bias	surrogate recoveries meet laboratory specified criteria	Surrogate Spike (organics)	A
		Sensitivity	Method specific criteria for calibration factors	Low Point Calibration Curve	A
		Completeness	90%	Completeness Check	A

Measurement Performance Criteria Table

Matrix	Groundwater/Surface Water
Analytical Group	SVOC
Concentration Level	Low

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)
URS Sampling Procedure	EPA Method 8270C/SOP-OA-1100-SVOC;(see Worksheet #23 for corresponding lab SOPs)	Precision	RPD=30	Field Duplicates	S & A
		Accuracy, Bias, Contamination	No target analytes above RL	Field Blanks, Laboratory Method Blanks	S & A
		Accuracy/Bias	+/- 30	LCS	A
		Accuracy/Bias	surrogate recoveries meet laboratory specific criteria	Surrogate Spike (organics)	A
		Sensitivity	Method specific criteria for calibration factors	Low Point Calibration Curve	A
		Completeness	>90%	Completeness Check	A

Measurement Performance Criteria Table

Matrix	Soil/Sediment
Analytical Group	SVOC
Concentration Level	Low

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)
URS Sampling Procedure	EPA Method 8270C//SOP-OA-1100-SVOC;(see Worksheet #23 for corresponding lab SOPs)	Precision	RPD=50	Field Duplicates	S & A
		Accuracy, Bias, Contamination	No target analytes above RL	Field Blanks, Laboratory Method Blanks	S & A
		Accuracy/Bias	+/- 30	LCS	A
		Accuracy/Bias	surrogate recoveries meet laboratory specific criteria	Surrogate Spike (organics)	A
		Sensitivity	Method specific criteria for calibration factors	Low Point Calibration Curve	A
		Completeness	>90%	Completeness Check	A

Measurement Performance Criteria Table

Matrix	Groundwater/Surface Water
Analytical Group	PCBs
Concentration Level	Low

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)
URS Sampling Procedure	EPA Method 8082A/SOP-OA-1500-PCBs; (see Worksheet #23 for corresponding lab SOPs)	Precision	RPD=30	Field Duplicates	S & A
		Accuracy, Bias, Contamination	No target analytes above RL	Field Blanks, Laboratory Method Blanks	S & A
		Accuracy/Bias	+/- 30	LCS	A
		Accuracy/Bias	surrogate recoveries meet laboratory specific criteria	Surrogate Spike (organics)	A
		Sensitivity	Method specific criteria for calibration factors	Low Point Calibration Curve	A
		Completeness	>90%	Completeness Check	A

Measurement Performance Criteria Table

Matrix	Soil/Sediment
Analytical Group	PCBs
Concentration Level	Low

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)
URS Sampling Procedure	EPA Method 8082A//SOP-OA-1500-PCB;(see Worksheet #23 for corresponding lab SOPs)	Precision	RPD=50	Field Duplicates	S & A
		Accuracy, Bias, Contamination	No target analytes above RL	Field Blanks, Laboratory Method Blanks	S & A
		Accuracy/Bias	+/- 30	LCS	A
		Accuracy/Bias	surrogate recoveries meet laboratory specific criteria	Surrogate Spike (organics)	A
		Sensitivity	Method specific criteria for calibration factors	Low Point Calibration Curve	A
		Completeness	>90%	Completeness Check	A

Measurement Performance Criteria Table

Matrix	Groundwater/Surface Water
Analytical Group	TAL Metals
Concentration Level	Low

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)
URS Sampling Procedure	EPA Method 6010B/7470/SOP-M-100-metals;(see Worksheet #23 for corresponding lab SOPs)	Precision	RPD=30	Field Duplicates	S & A
		Precision	RPD=30	Laboratory Duplicates	A
		Accuracy, Bias, Contamination	No target analytes above RL	Field Blanks, Laboratory Method Blanks	S & A
		Accuracy/Bias	75-125; Hg=80-110	LCS	A
		Accuracy/Bias	75-125; Hg=80-110	Matrix Spike (inorganics)	A
		Sensitivity	Method specific criteria for calibration factors	Low Point Calibration Curve	A
		Completeness	>90%	Completeness Check	A

Measurement Performance Criteria Table

Matrix	Soil/Sediment
Analytical Group	TAL Metals
Concentration Level	Low

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)
URS Sampling Procedure	EPA Method 6010B/7470/SOP-1000-M-metals; (see Worksheet #23 for corresponding lab SOPs)	Precision	RPD=30	Field Duplicates	S & A
		Precision	RPD=30	Laboratory Duplicates	A
		Accuracy, Bias, Contamination	No target analytes above RL	Field Blanks, Laboratory Method Blanks	S & A
		Accuracy/Bias	75-125; Hg=80-110	LCS	A
		Accuracy/Bias	75-125; Hg=80-110	Matrix Spike (inorganics)	A
		Sensitivity	Method specific criteria for calibration factors	Low Point Calibration Curve	A
		Completeness	>90%	Completeness Check	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #13

(UFP-QAPP Manual Section 2.7)

Identify all secondary data and information that will be used for the project and their originating sources. Specify how the secondary data will be used and the limitations on their use.

☑ Worksheet Not Applicable (State Reason) **not applicable to this phase of the project.** **See Worksheet #10 for further information.**

Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use

QAPP Worksheet #14

(UFP-QAPP Manual Section 2.8.1)

Provide a brief overview of the listed project activities.

☐ Worksheet Not Applicable (State Reason)

Summary of Project Tasks

Sampling Tasks: Quarterly groundwater from monitoring wells at the site. Collection of sediment and surface water samples will be determined at a later date and the collection will be on an as need basis.
Analysis Tasks: Groundwater samples will be analyzed for VOCs and SVOCs. Sediment samples will be analyzed for VOCs, SVOCs, PCBs, and metals.
Quality Control Tasks: Field personnel will collect QC samples including field blanks for all parameters and trip blanks for VOCs only, additional sample for MS/MSD, and field duplicates at the rate of one per 20 samples. Laboratory QC will include the use of surrogate compounds for VOCs, SVOCs and PCBs, LCS, mass spectral tune, internal stardards, initial and continuing calibration verification and laboratory method blanks. Metals QC will include initial and continuing calibration, laboratory duplicate, MS, interference check sample.
Secondary Data: Field personnel will measure parameters in the field including pH, temperature, conductivity.
Data Management Tasks: All data generated through field activities or by laboratory analysis shall be reduced and reviewed before being reported. Samples will be collected by field personnel and sent by common carrier with the completed chain of custody form to the laboratory. Reports of analytical data will be sent by the laboratory to the URS Project Manager, URS QA Officer, and the Detrex Project Manager. URS and STL will attempt to minimize manual data entry through the use of direct electronic transfer from the laboratory instrumentation to the laboratory information management system (LIMS) and the use of data software packages. The laboratory will transmit reviewed data to URS electronically. URS will review all data supplied by the laboratory and the laboratory reported data will be checked to see that all QC criteria are met and the data meets the DQOs for the investigation. Once data are reviewed and validated, they will be stored in a database. Data will also be transmitted electronically by the laboratory to a project database maintained by URS for Detrex. Data deliverables of all analytical data for the project will be stored on CD in as pdf files and maintained in the file maintained by the URS Project Manager. Data deliverables will be provided to US EPA Region 5 on CD in pdf format.
Documentation and Records: The laboratory will general a level 4 data package that will include chain of custody, airbills, and sample logs and will be stored in the file at URS Corp. along with field notebooks.
Assessment/Audit Tasks: Project safety analysis will be conducted for all sampling tasks and a field audit may be performed by the URS Health and Safety officer. A laboratory audit is not anticipated during the sampling unless requested by US EPA Region 5.

Data Review Tasks: The laboratory will verify that all data are complete for samples received. The URS QA officer will validate the data using EPA Functional Guidelines for the Review of Organic and Inorganic Data as a guideline for evaluating the data. Since the Functional Guidelines deal mainly with CLP data other guidance documents including the US EPA Region 2 data validation guildelines listed below, which are specific to SW-846 methods. These include:

- US EPA Region 2 SOP HW-22 for SW-846 Method 8270C (SVOCs)(6/2001)
- US EPA Region 2 SOP HW-23B for SW-846 Method 8082 (PCBs)(6/1999)
- US EPA Region 2 SOP HW-24 for SW-846 Method 8260B (VOCs)(4/1999)

In addition laboratory specific QC criteria from Firstechnology's QAPP and individual method SOPs mentioned in Worksheet #23 will also be used to evaluate the data. URS will evaluate all project specific measurement performance criteria and data validation criteria. A data usability summary will be produced for submittal with the data.

Validaed data and all field logs/notebooks and notes will be reviewed to assess total measurement eror and determine oveall usability of the data. for project purposes. Data limitations will be determined and data will be compared to project DQOs and against clean up action levels. Final data will be stored in a database, with any necessary qualifiers and notes that are generated.

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Finally, list the published and achievable detection and quantitation limits for each analyte.

☐ Worksheet Not Applicable (State Reason)

Reference Limits and Evaluation Table

Matrix: Groundwater/Surface Water

Analytical Group: VOA

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (ug/l)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
1,1,1,2-Tetrachloroethane	630-20-6	NA	5	1.92	5	1.92	5
1,1,1-Trichloroethane	71-55-6	NA	5	2.62	5	2.62	5
1,1,2,2-Tetrachloroethane	79-34-5	NA	5	3.33	5	3.33	5
1,1,2-Trichloroethane	79-00-5	NA	5	3.32	5	3.32	5
1,1-Dichloroethane	75-34-3	NA	5	2.8	5	2.8	5
1,1-Dichloroethene	75-35-4	NA	5	3.28	5	3.28	5
1,1-Dichloropropene	563-58-6	NA	5	1.87	5	1.87	5
1,2,3-Trichloropropane	96-18-4	NA	5	1.73	5	1.73	5
1,2,4-Trimethylbenzene	95-63-6	NA	5	2.03	5	2.03	5
1,2-Dibromo-3-chloropropane	96-12-8	NA	20	17.5	20	17.5	20
1,2-Dibromoethane	106-93-4	NA	5	2.4	5	2.4	5
1,2-Dichlorobenzene	95-50-1	NA	5	2.61	5	2.61	5
1,2-Dichloroethane	107-06-2	NA	5	2.75	5	2.75	5
1,2-Dichloropropane	78-87-5	NA	5	1.52	5	1.52	5
1,3,5-Trimethylbenzene	108-67-8	NA	5	1.83	5	1.83	5
1,3-Dichlorobenzene	541-73-1	NA	5	1.78	5	1.78	5
1,4-Dichlorobenzene	106-46-7	NA	5	1.32	5	1.32	5
2,2-Dichloropropane	594-20-7	NA	5	2.52	5	2.52	5

Reference Limits and Evaluation Table

Matrix: Groundwater/Surface Water

Analytical Group: VOA

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (ug/l)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
2-Butanone	78-93-3	NA	50	5.74	50	5.74	50
2-Chlorotoluene	95-49-8	NA	5	2.47	5	2.47	5
2-Hexanone	591-78-6	NA	5	6.06	5	6.06	5
4-Chlorotoluene	106-43-4	NA	5	2.26	5	2.26	5
4-Methyl-2-pentanone	108-10-1	NA	50	3.39	50	3.39	50
Acetone	67-64-1	NA	100	6.86	100	6.86	5
Benzene	71-43-2	NA	5	2.15	5	2.15	5
Bromobenzene	108-86-1	NA	5	1.45	5	1.45	5
Bromodichloromethane	75-27-4	NA	5	2.65	5	2.65	5
Bromoform	75-25-2	NA	5	1.89	5	1.89	5
Bromomethane	74-83-9	NA	10	1.63	10	1.63	10
Carbon disulfide	75-15-0	NA	5	5.28	5	5.28	5
Carbon tetrachloride	56-23-5	NA	5	2.15	5	2.15	5
Chlorobenzene	108-90-7	NA	5	1.44	5	1.44	5
Chlorodibromomethane	124-48-1	NA	5	2.46	5	2.46	5
Chloroethane	75-00-3	NA	10	2.87	10	2.87	10
Chloroform	67-66-3	NA	5	2.43	5	2.43	5
Chloromethane	74-87-3	NA	10	3.03	10	3.03	10
cis-1,2-Dichloroethene	156-59-2	NA	5	1.95	5	1.95	5
cis-1,3-Dichloropropene	10061-01-5	NA	5	2.89	5	2.89	5
Dibromomethane	74-95-3	NA	5	2.98	5	2.98	5
Dichlorodifluoromethane	75-71-8	NA	20	3.13	20	3.13	20
Ethylbenzene	100-41-4	NA	5	1.82	5	1.82	5
Isopropylbenzene	98-82-8	NA	5	1.83	5	1.83	5
m,p-Xylene	108-38-3/106-42-3	NA	10	4.88	10	4.88	10
Methyl tert-butyl ether	1634-04-4	NA	5	2.68	5	2.68	5
Methylene chloride	75-09-2	NA	5	5.16	5	5.16	5
Naphthalene	91-20-3	NA	5	na	5	na	5

Reference Limits and Evaluation Table

Matrix: Groundwater/Surface Water

Analytical Group: VOA

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (ug/l)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
n-Butylbenzene	104-51-8	NA	5	1.48	5	1.48	5
n-Propylbenzene	103-65-1	NA	5	2.47	5	2.47	5
o-Xylene	95-47-6	NA	5	1.44	5	1.44	5
p-Isopropyltoluene	99-87-6	NA	5	1.32	5	1.32	5
sec-Butylbenzene	135-98-8	NA	5	2.03	5	2.03	5
Styrene	100-42-5	NA	5	2.42	5	2.42	5
tert-Butylbenzene	98-06-6	NA	5	1.32	5	1.32	5
Tetrachloroethene	127-18-4	NA	5	2.32	5	2.32	5
Toluene	108-88-3	NA	5	2.89	5	2.89	5
trans-1,2-Dichloroethene	156-60-5	NA	5	2.45	5	2.45	5
trans-1,3-Dichloropropene	10061-02-6	NA	5	2.42	5	2.42	5
Trichloroethene	79-01-6	NA	5	2.39	5	2.39	5
Trichlorofluoromethane	75-69-4	NA	5	2.39	5	2.39	5
Vinyl acetate	108-05-4	NA	50	6.23	50	6.23	50
Vinyl chloride	75-01-4	NA	2	2.97	2	2.97	2
Xylene (total)	1330-20-7	NA	15	6.32	15	6.32	15

Reference Limits and Evaluation Table

Matrix: Soil/Sediment

Analytical Group: VOA

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (ug/kg)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
1,1,1,2-Tetrachloroethane	630-20-6	NA	5	1.92	5	1.92	5
1,1,1-Trichloroethane	71-55-6	NA	5	2.62	5	2.62	5
1,1,2,2-Tetrachloroethane	79-34-5	NA	5	3.33	5	3.33	5
1,1,2-Trichloroethane	79-00-5	NA	5	3.32	5	3.32	5
1,1-Dichloroethane	75-34-3	NA	5	2.8	5	2.8	5
1,1-Dichloroethene	75-35-4	NA	5	3.28	5	3.28	5
1,1-Dichloropropene	563-58-6	NA	5	1.87	5	1.87	5
1,2,3-Trichloropropane	96-18-4	NA	5	1.73	5	1.73	5
1,2,4-Trimethylbenzene	95-63-6	NA	5	2.03	5	2.03	5
1,2-Dibromo-3-chloropropane	96-12-8	NA	20	17.5	20	17.5	20
1,2-Dibromoethane	106-93-4	NA	5	2.4	5	2.4	5
1,2-Dichlorobenzene	95-50-1	NA	5	2.61	5	2.61	5
1,2-Dichloroethane	107-06-2	NA	5	2.75	5	2.75	5
1,2-Dichloropropane	78-87-5	NA	5	1.52	5	1.52	5
1,3,5-Trimethylbenzene	108-67-8	NA	5	1.83	5	1.83	5
1,3-Dichlorobenzene	541-73-1	NA	5	1.78	5	1.78	5
1,4-Dichlorobenzene	106-46-7	NA	5	1.32	5	1.32	5
2,2-Dichloropropane	594-20-7	NA	5	2.52	5	2.52	5
2-Butanone	78-93-3	NA	50	5.74	50	5.74	50
2-Chlorotoluene	95-49-8	NA	5	2.47	5	2.47	5
2-Hexanone	591-78-6	NA	5	6.06	5	6.06	5
4-Chlorotoluene	106-43-4	NA	5	2.26	5	2.26	5
4-Methyl-2-pentanone	108-10-1	NA	50	3.39	50	3.39	50
Acetone	67-64-1	NA	100	6.86	100	6.86	5
Benzene	71-43-2	NA	5	2.15	5	2.15	5

Reference Limits and Evaluation Table

Matrix: Soil/Sediment

Analytical Group: VOA

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (ug/kg)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
Bromobenzene	108-86-1	NA	5	1.45	5	1.45	5
Bromodichloromethane	75-27-4	NA	5	2.65	5	2.65	5
Bromoform	75-25-2	NA	5	1.89	5	1.89	5
Bromomethane	74-83-9	NA	10	1.63	10	1.63	10
Carbon disulfide	75-15-0	NA	5	5.28	5	5.28	5
Carbon tetrachloride	56-23-5	NA	5	2.15	5	2.15	5
Chlorobenzene	108-90-7	NA	5	1.44	5	1.44	5
Chlorodibromomethane	124-48-1	NA	5	2.46	5	2.46	5
Chloroethane	75-00-3	NA	10	2.87	10	2.87	10
Chloroform	67-66-3	NA	5	2.43	5	2.43	5
Chloromethane	74-87-3	NA	10	3.03	10	3.03	10
cis-1,2-Dichloroethene	156-59-2	NA	5	1.95	5	1.95	5
cis-1,3-Dichloropropene	10061-01-5	NA	5	2.89	5	2.89	5
Dibromomethane	74-95-3	NA	5	2.98	5	2.98	5
Dichlorodifluoromethane	75-71-8	NA	20	3.13	20	3.13	20
Ethylbenzene	100-41-4	NA	5	1.82	5	1.82	5
Isopropylbenzene	98-82-8	NA	5	1.83	5	1.83	5
m,p-Xylene	108-38-3/106-42-3	NA	10	4.88	10	4.88	10
Methyl tert-butyl ether	1634-04-4	NA	5	2.68	5	2.68	5
Methylene chloride	75-09-2	NA	5	5.16	5	5.16	5
Naphthalene	91-20-3	NA	5	na	5	na	5
n-Butylbenzene	104-51-8	NA	5	1.48	5	1.48	5
n-Propylbenzene	103-65-1	NA	5	2.47	5	2.47	5
o-Xylene	95-47-6	NA	5	1.44	5	1.44	5
p-Isopropyltoluene	99-87-6	NA	5	1.32	5	1.32	5
sec-Butylbenzene	135-98-8	NA	5	2.03	5	2.03	5
Styrene	100-42-5	NA	5	2.42	5	2.42	5
tert-Butylbenzene	98-06-6	NA	5	1.32	5	1.32	5
Tetrachloroethene	127-18-4	NA	5	2.32	5	2.32	5

Reference Limits and Evaluation Table

Matrix: Soil/Sediment

Analytical Group: VOA

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (ug/kg)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
Toluene	108-88-3	NA	5	2.89	5	2.89	5
trans-1,2-Dichloroethene	156-60-5	NA	5	2.45	5	2.45	5
trans-1,3-Dichloropropene	10061-02-6	NA	5	2.42	5	2.42	5
Trichloroethene	79-01-6	NA	5	2.39	5	2.39	5
Trichlorofluoromethane	75-69-4	NA	5	2.39	5	2.39	5
Vinyl acetate	108-05-4	NA	50	6.23	50	6.23	50
Vinyl chloride	75-01-4	NA	2	2.97	2	2.97	2
Xylene (total)	1330-20-7	NA	15	6.32	15	6.32	15

Reference Limits and Evaluation Table

Matrix: Groundwater/Surface Water

Analytical Group: SVOC

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (ug/l)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
1,2,4-Trichlorobenzene	120-82-1	NA	10	7	10	7	10
1,2-Dichlorobenzene	95-50-1	NA	10	1.7	10	1.7	10
1,3-Dichlorobenzene	541-73-1	NA	10	1.8	10	1.8	10
1,4-Dichlorobenzene	106-46-7	NA	10	1.34	10	1.34	10
1-Methylnaphthalene	90-12-0	NA	10	NA	10	NA	10
2,3,4,6-Tetrachlorophenol	58-90-2	NA	10	NA	10	NA	10
2,4,5-Trichlorophenol	95-95-4	NA	10	2.3	10	2.3	10
2,4,6-Trichlorophenol	88-06-2	NA	10	2.2	10	2.2	10
2,4-Dichlorophenol	120-83-2	NA	10	2.4	10	2.4	10
2,4-Dimethylphenol	105-67-9	NA	10	2.6	10	2.6	10
2,4-Dinitrophenol	51-28-5	NA	50	4.9	50	4.9	50
2,4-Dinitrotoluene	121-14-2	NA	10	2	10	2	10
2,6-Dinitrotoluene	606-20-2	NA	10	3.2	10	3.2	10
2-Chloronaphthalene	91-58-7	NA	10	7	10	7	10
2-Chlorophenol	95-57-8	NA	10	3.3	10	3.3	10
2-Methylnaphthalene	91-57-6	NA	10	6.2	10	6.2	10
2-Methylphenol	95-48-7	NA	10	3.3	10	3.3	10
2-Naphthylamine	91-59-8	NA	10	NA	10	NA	10
2-Nitroaniline	88-74-4	NA	10	3.9	10	3.9	10
2-Nitrophenol	88-75-5	NA	10	2.8	10	2.8	10
3,3'-Dichlorobenzidine	91-94-1	NA	20	2	20	2	20
3-Methylphenol/4-Methylphenol	106-44-5	NA	20	3.3	20	3.3	20
3-Nitroaniline	99-09-2	NA	10	6.1	10	6.1	10
4,6-Dintro-2-methylphenol	534-52-1	NA	50	2.9	50	2.9	50
4-Bromophenyl phenyl ether	101-55-3	NA	10	6.2	10	6.2	10

Reference Limits and Evaluation Table

Matrix: Groundwater/Surface Water

Analytical Group: SVOC

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (ug/l)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
4-Chloro-3-methylphenol	59-50-7	NA	50	3.4	50	3.4	50
4-Chloroaniline	106-47-8	NA	10	7.5	10	7.5	10
4-Chlorophenyl phenyl ether	7005-72-3	NA	10	7.3	10	7.3	10
4-Nitroaniline	100-01-6	NA	10	6.1	10	6.1	10
4-Nitrophenol	100-02-7	NA	50	1.7	50	1.7	50
Acenaphthene	83-32-9	NA	20	6	20	6	20
Acenaphthylene	208-96-8	NA	10	5.3	10	5.3	10
Aniline	62-53-3	NA	10	5.7	10	5.7	10
Anthracene	120-12-7	NA	10	3.6	10	3.6	10
Benz(a)anthracene	56-55-3	NA	10	1.4	10	1.4	10
Benzidine	92-87-5	NA	10	4.19	10	4.19	10
Benzo(a)pyrene	50-32-8	NA	10	1.8	10	1.8	10
Benzo(b)fluoranthene	205-99-2	NA	20	2	20	2	20
Benzo(g,h,i)perylene	191-24-2	NA	10	1.5	10	1.5	10
Benzo(k)fluoranthene	207-08-9	NA	10	2	10	2	10
Benzoic acid	65-85-0	NA	10	1.1	10	1.1	10
Benzyl alcohol	100-51-6	NA	10	3.1	10	3.1	10
Bis(2-chlorethoxy)methane	111-91-1	NA	10	NA	10	NA	10
Bis(2-chloroethyl) ether	111-44-4	NA	10	2.5	10	2.5	10
Bis(2-chloroethyl) ether	111-44-4	NA	10	2.5	10	2.5	10
Bis(2-chloroisopropyl) ether	108-60-1	NA	10	5.6	10	5.6	10
Bis(2-ethylhexyl) phthalate	117-81-7	NA	10	2.6	10	2.6	10
Butyl benzyl phthalate	85-68-7	NA	10	1.7	10	1.7	10
Carbazole	86-74-8	NA	10	NA	10	NA	10
Chrysene	218-01-9	NA	10	1.3	10	1.3	10

Reference Limits and Evaluation Table

Matrix: Groundwater/Surface Water

Analytical Group: SVOC

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (ug/l)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
Dibenz(a,h)anthracene	53-70-3	NA	10	1.1	10	1.1	10
Dibenzofuran	132-64-9	NA	10	4.5	10	4.5	10
Diethyl phthalate	84-66-2	NA	10	2.6	10	2.6	10
Dimethyl phthalate	131-11-3	NA	10	3.9	10	3.9	10
Di-n-butyl phthalate	84-74-2	NA	10	2.6	10	2.6	10
Di-n-octyl phthalate	117-84-0	NA	10	2.7	10	2.7	10
Diphenlyamine	122-39-4	NA	10	NA	10	NA	10
Fluoranthene	206-44-0	NA	10	1.9	10	1.9	10
Fluorene	86-73-7	NA	10	5.2	10	5.2	10
Hexachlorobenzene	118-74-1	NA	10	7.2	10	7.2	10
Hexachlorobutadiene	87-68-3	NA	10	7.1	10	7.1	10
Hexachlorocyclopentadiene	77-47-4	NA	10	7.1	10	7.1	10
Hexachloroethane	67-72-1	NA	10	6.8	10	6.8	10
Indeno(1,2,3-cd)pyrene	193-39-5	NA	10	4.6	10	4.6	10
Isophorone	78-59-1	NA	10	2.8	10	2.8	10
Naphthalene	91-20-3	NA	10	5.8	10	5.8	10
N-Nitrosodimethylamine	62-75-9	NA	10	1.7	10	1.7	10
N-Nitrosodi-n-propylamine	621-64-7	NA	10	3.4	10	3.4	10
N-Nitrosodiphenylamine	86-30-6	NA	10	2.9	10	2.9	10
Pentachlorophenol	87-86-5	NA	10	5.4	10	5.4	50
Phenanthrene	85-01-8	NA	10	3.5	10	3.5	10
Phenol	108-95-2	NA	10	2.8	10	2.8	10
Pyrene	129-00-0	NA	10	1.9	10	1.9	10
Pyridine	110-86-1	NA	10	2.9	10	2.9	10

Reference Limits and Evaluation Table

Matrix: Soil/Sediments

Analytical Group: SVOC

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (applicable units)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
1,2,4-Trichlorobenzene	120-82-1	NA	330	233	330	233	330
1,2-Dichlorobenzene	95-50-1	NA	330	56	330	56	330
1,2-Diphenylhydrazine	122-66-7	NA	330	111	330	111	330
1,3-Dichlorobenzene	541-73-1	NA	330	58	330	58	330
1,4-Dichlorobenzene	106-46-7	NA	330	45	330	45	330
1,4-Dioxane	123-91-1	NA	330	NA	330	NA	330
1-Methylnaphthalene	90-12-0	NA	330	NA	330	NA	330
2,3,4,6-Tetrachlorophenol	58-90-2	NA	330	NA	330	NA	330
2,4,5-Trichlorophenol	95-95-4	NA	330	75	330	75	330
2,4,6-Tribromophenol	118-79-6	NA	330	73	330	73	330
2,4-Dichlorophenol	120-83-2	NA	330	80	330	80	330
2,4-Dimethylphenol	105-67-9	NA	330	87	330	87	330
2,4-Dinitrophenol	51-28-5	NA	1700	162	1700	162	1700
2,6-Dinitrotoluene	606-20-2	NA	330	67	330	67	330
2-Chloronaphthalene	91-58-7	NA	330	233	330	233	330
2-Chlorophenol	95-57-8	NA	330	110	330	110	330
2-Methylnaphthalene	91-57-6	NA	330	207	330	207	330
2-Methylphenol	95-48-7	NA	330	110	330	110	330
2-Naphthylamine	91-59-8	NA	330	NA	330	NA	330
2-Nitroaniline	88-74-4	NA	330	130	330	130	330
2-Nitrophenol	88-75-5	NA	330	93	330	93	330
3,3'-Dichlorobenzidine	91-94-1	NA	330	NA	330	NA	330
3-Methylphenol/4-Methyl phenol	106-44-5	NA	670	110	670	110	670
3-Nitroaniline	99-09-2	NA	330	203	330	203	330
4,6-Dintro-2-methylpheno l	534-52-1	NA	1700	97	1700	97	1700
4-Bromophenyl phenyl ether	101-55-3	NA	330	207	330	207	330

Reference Limits and Evaluation Table

Matrix: Soil/Sediments

Analytical Group: SVOC

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (applicable units)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
4-Chloro-3-methylphenol	59-50-7	NA	1700	113	1700	113	1700
4-Chloroaniline	106-47-8	NA	330	249	330	249	330
4-Chlorophenyl phenyl ether	7005-72-3	NA	330	243	330	243	330
4-Nitroaniline	100-01-6	NA	330	203	330	203	330
4-Nitrophenol	100-02-7	NA	1700	57	1700	57	1700
Acenaphthene	83-32-9	NA	330	200	330	200	330
Acenaphthylene	208-96-8	NA	330	177	330	177	330
Aniline	62-53-3	NA	330	190	330	190	330
Anthracene	120-12-7	NA	330	120	330	120	330
Benz(a)anthracene	56-55-3	NA	330	47	330	47	330
Benzidine	92-87-5	NA	330	140	330	140	330
Benzo(a)pyrene	50-32-8	NA	330	60	330	60	330
Benzo(b)fluoranthene	205-99-2	NA	330	67	330	67	330
Benzo(g,h,i)perylene	191-24-2	NA	330	50	330	50	330
Benzo(k)fluoranthene	207-08-9	NA	330	67	330	67	330
Benzoic acid	65-85-0	NA	330	37	330	37	330
Benzyl alcohol	100-51-6	NA	330	103	330	103	330
Bis(2-chlorethoxy)methane	111-91-1	NA	330	NA	330	NA	330
Bis(2-chloroethyl) ether	111-44-4	NA	330	83	330	83	330
Bis(2-chloroisopropyl) ether	108-60-1	NA	330	186	330	186	330
Bis(2-ethylhexyl) phthalate	117-81-7	NA	330	87	330	87	330
Butyl benzyl phthalate	85-68-7	NA	330	57	330	57	330
Carbazole	86-74-8	NA	330	NA	330	NA	330
Chrysene	218-01-9	NA	330	43	330	43	330
Dibenz(a,h)anthracene	53-70-3	NA	330	37	330	37	330
Dibenzofuran	132-64-9	NA	330	150	330	150	330

Reference Limits and Evaluation Table

Matrix: Soil/Sediments

Analytical Group: SVOC

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (applicable units)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
Diethyl phthalate	84-66-2	NA	330	87	330	87	330
Dimethyl phthalate	131-11-3	NA	330	130	330	130	330
Di-n-butyl phthalate	84-74-2	NA	330	NA	330	NA	330
Di-n-octyl phthalate	117-84-0	NA	330	90	330	90	330
Diphenlyamine	122-39-4	NA	330	NA	330	NA	330
Fluoranthene	206-44-0	NA	330	63	330	63	330
Fluorene	86-73-7	NA	330	173	330	173	330
Hexachlorobenzene	118-74-1	NA	330	240	330	240	330
Hexachlorobutadiene	87-68-3	NA	330	237	330	237	330
Hexachlorocyclopentadiene	77-47-4	NA	330	60	330	60	330
Hexachloroethane	67-72-1	NA	330	227	330	227	330
Indeno(1,2,3-cd)pyrene	193-39-5	NA	330	153	330	153	330
Isophorone	78-59-1	NA	330	93	330	93	330
Naphthalene	91-20-3	NA	330	193	330	193	330
Nitrobenzene	98-95-3	NA	330	133	330	133	330
N-Nitrosodimethylamine	62-75-9	NA	330	57	330	57	330
N-Nitrosodi-n-propylamine	621-64-7	NA	330	113	330	113	330
N-Nitrosodiphenylamine	86-30-6	NA	330	97	330	97	330
Pentachlorophenol	87-86-5	NA	1700	180	1700	180	1700
Phenanthrene	85-01-8	NA	330	117	330	117	330
Phenol	108-95-2	NA	330	93	330	93	330
Pyrene	129-00-0	NA	330	63	330	63	330
Pyridine	110-86-1	NA	330	63	330	63	330

Reference Limits and Evaluation Table

Matrix: Groundwater/Surface Water

Analytical Group: PCBs

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (ug/l)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
Aroclor 1016	12674-11-2	NA	0.25	0.018	0.25	0.018	0.25
Aroclor 1221	11104-28-2	NA	0.50	0.026	0.50	0.026	0.50
Aroclor 1232	11141-16-5	NA	0.50	0.024	0.50	0.024	0.50
Aroclor 1242	53469-21-9	NA	0.25	0.033	0.25	0.033	0.25
Aroclor 1248	12672-29-6	NA	0.25	0.036	0.25	0.036	0.25
Aroclor 1254	11097-69-1	NA	0.25	0.021	0.25	0.021	0.25
Aroclor 1260	11096-82-5	NA	0.25	0.026	0.25	0.026	0.25

Reference Limits and Evaluation Table

Matrix: Soil/Sediments

Analytical Group: PCBs

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (mg/kg	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
Aroclor 1016	12674-11-2		0.13	0.009	0.13	0.009	0.13
Aroclor 1221	11104-28-2		0.25	0.018	0.25	0.018	0.25
Aroclor 1232	11141-16-5		0.25	0.012	0.25	0.012	0.25
Aroclor 1242	53469-21-9		0.13	0.016	0.13	0.016	0.13
Aroclor 1248	12672-29-6		0.13	0.018	0.13	0.018	0.13
Aroclor 1254	11097-69-1		0.13	0.010	0.13	0.010	0.13
Aroclor 1260	11096-82-5		0.13	0.013	0.13	0.013	0.13

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: TAL Metals/Cyanide

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (ug/l)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
Aluminum	7429-90-5	NA	100	21	100	21	100
Antimony	7440-36-0	NA	100	26	100	26	100
Arsenic	7440-38-2	NA	100	45	100	45	100
Barium	7440-39-3	NA	5	8	5	8	5
Beryllium	7440-41-7	NA	10	0.92	10	0.92	10
Cadmium	7440-43-9	NA	10	4.9	10	4.9	10
Calcium	7440-70-2	NA	50	4.8	50	4.8	50
Chromium	7440-47-3	NA	10	4.8	10	4.8	10
Cobalt	7440-48-4	NA	10	9.1	10	9.1	10
Copper	7440-50-8	NA	10	4.4	10	4.4	10
Iron	7439-89-6	NA	20	13	20	13	20
Lead	7439-92-1	NA	20	5.2	20	5.2	20
Magnesium	7439-95-4	NA	20	2.4	20	2.4	20
Manganese	7439-96-5	NA	10	10	10	10	10
Mercury	7439-97-6	NA	0.2	0.13	0.2	0.13	0.2
Nickel	7440-02-0	NA	10	6.8	10	6.8	10
Potassium	7440-09-7	NA	20	12	20	12	20
Selenium	7782-49-2	NA	100	44	100	44	100
Silver	7440-22-4	NA	10	4.3	10	4.3	10
Sodium	64742-46-7	NA	20	11	20	11	20
Thallium	7440-28-0	NA	100	42	100	42	100
Vanadium	7440-62-2	NA	10	4.6	10	4.6	10

Reference Limits and Evaluation Table

Matrix: Soil

Analytical Group: TAL Metals/Cyanide

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit (mg/kg)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
Aluminum	7429-90-5	NA	5	NA	5	NA	5
Antimony	7440-36-0	NA	5	NA	5	NA	5
Arsenic	7440-38-2	NA	5	NA	5	NA	5
Barium	7440-39-3	NA	0.25	NA	0.25	NA	0.25
Beryllium	7440-41-7	NA	0.5	NA	0.5	NA	0.5
Cadmium	7440-43-9	NA	0.5	NA	0.5	NA	0.5
Calcium	7440-70-2	NA	2.5	NA	2.5	NA	2.5
Chromium	7440-47-3	NA	0.5	NA	0.5	NA	0.5
Cobalt	7440-48-4	NA	0.5	NA	0.5	NA	0.5
Copper	7440-50-8	NA	0.5	NA	0.5	NA	0.5
Iron	7439-89-6	NA	1	NA	1	NA	1
Lead	7439-92-1	NA	1	NA	1	NA	1
Magnesium	7439-95-4	NA	1	NA	1	NA	1
Manganese	7439-96-5	NA	0.5	NA	0.5	NA	0.5
Mercury	7439-97-6	NA	0.01	NA	0.01	NA	0.01
Nickel	7440-02-0	NA	0.5	NA	0.5	NA	0.5
Potassium	7440-09-7	NA	1	NA	1	NA	1
Selenium	7782-49-2	NA	5	NA	5	NA	5
Silver	7440-22-4	NA	1	NA	1	NA	1
Sodium	64742-46-7	NA	1	NA	1	NA	1
Thallium	7440-28-0	NA	5	NA	5	NA	5
Vanadium	7440-62-2	NA	0.5	NA	0.5	NA	0.5

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QAPP Worksheet #16

(UFP-QAPP Manual Section 2.8.2)

List all project activities as well as the QA assessments that will be performed during the course of the project. Include the anticipated start and completion dates.

☐ Worksheet Not Applicable (State Reason)

Project Schedule Timeline Table

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Monitoring Well Groundwater Sampling	URS Corporation	Ongoing		Groundwater Monitoring Report	Quarterly
Monitoring Well Groundwater Sampling Data Review	URS Corporation	TBD	TBD	Data Review Summary	Quarterly
Trench Groundwater Sampling	URS Corporation	March 15, 2007	TBD	Initial Report	June 2007
Trench Groundwater Sampling Data Review	URS Corporation	May 1, 2007	TBD	Data Review Summary	June 2007
DNAPL Well Installation	URS Corporation	May 2007	TBD	Summary Report	August 2007
DS Sediment Sampling	URS Corporation	Spring 2007	TBD	Summary Report	Summer 2007
DS Sediment Sampling Data Review	URS Corporation	Spring 2007	TBD	Data Review Summary Report	Summer 2007

QAPP Worksheet #17

(UFP-QAPP Section 3.1.1)

Describe the project sampling approach. Provide the rationale for selecting sample locations and matrices for each analytical group and concentration level.

☐ Worksheet Not Applicable (State Reason)

Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):
Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [May refer to map or Worksheet #18 for details]: *Groundwater samples will be collected at the site from existing monitoring wells and measured for VOCs and SVOCs using EPA Methods 8260 and 8270. Standard concentrations will be used as the reporting limits. These concentrations are listed in QAPP Worksheet #15. Field Sampling personnel will collect three extra samples at one location per 20 samples for matrix spike/matrix spike duplicate; One duplicate sample will be collected per 20 samples that will be blind to the laboratory. Sampling personnel will also collect one field duplicate sample per day. Sediment samples will be collected at locations to be determined at a later date. The same analytical parameters and QC frequency will be used for sediment samples.*

QAPP Worksheet #18

(UFP-QAPP Manual Section 3.1.1)

List all site locations that will be sampled and include sample/ID number, if available. (Provide a range of sampling locations or ID numbers if a site has a large number.) Specify matrix and, if applicable, depth at which samples will be taken. Only a short reference for the sampling location rationale is necessary for the table. The text of the QAPP should clearly identify the detailed rationale associated with each reference. Complete all required information, using additional worksheets if necessary.

☐ Worksheet Not Applicable (State Reason)

Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number	Matrix	Depth ( )	Analytical Group	Concentration Level	Number of Samples (identify field duplicates	Sampling SOP Reference[1]	Rationale for Sampling Location
DPT-01	Water	NA	VOCs, SVOCs	Low	1	Detrex Workplan (in progress)	Existing monitoring well
DPT-02	Water	NA	VOCs, SVOCs	Low	2 – field duplicate	Detrex Workplan (in progress)	Existing monitoring well
DPT-05	Water	NA	VOCs, SVOCs	Low	1	Detrex Workplan (in progress)	Existing monitoring well
DPT-06	Water	NA	VOCs, SVOCs	Low	1	Detrex Workplan (in progress)	Existing monitoring well
DPT-07	Water	NA	VOCs, SVOCs	Low	1	Detrex Workplan (in progress)	Existing monitoring well
DPT-10	Water	NA	VOCs, SVOCs	Low	1	Detrex Workplan (in progress)	Existing monitoring well
DPT-12	Water	NA	VOCs, SVOCs	Low	3 – MS/MSD	Detrex Workplan (in progress)	Existing monitoring well
DPT-13	Water	NA	VOCs, SVOCs	Low	1	Detrex Workplan (in progress)	Existing monitoring well
DPT-16	Water	NA	VOCs, SVOCs	Low	1	Detrex Workplan (in progress)	Existing monitoring well
DPT-19	Water	NA	VOCs, SVOCs	Low	1	Detrex Workplan (in progress)	Existing monitoring well
DPT-20	Water	NA	VOCs, SVOCs	Low	1	Detrex Workplan (in progress)	Existing monitoring well
DPT-22	Water	NA	VOCs, SVOCs	Low	1	Detrex Workplan (in progress)	Existing monitoring well
DPT-25	Water	NA	VOCs, SVOCs	Low	1	Detrex Workplan (in progress)	Existing monitoring well

Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number	Matrix	Depth ( )	Analytical Group	Concentration Level	Number of Samples (identify field duplicates	Sampling SOP Reference[1]	Rationale for Sampling Location
DPT-SED-	Sediment	NA	VOCs, SVOCs, metals	TBA	TBA	Detrex Workplan (in progress)	Determine extent of contaminatio n

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #21).

QAPP Worksheet #19

(UFP-QAPP Manual Section 3.1.1)

For each matrix, analytical group, and concentration level, list the analytical and preparation method/SOP and associated sample volume, container specifications, preservation requirements, and maximum holding time.

☐ Worksheet Not Applicable (State Reason)

Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference [1]	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Groundwater	VOCs	Low	EPA Method 5030/8260B	40 ml	3 x 40 ml VOA vials with Teflon® lined caps.	1:1 HCl to pH<2; 4°C	14 days
Groundwater	SVOCs	Low	EPA Method/8270C	1,000 ml	2 X 1,000 ml amber glass	Unpreserved; 4°C	7 days to extraction; 40 days from extraction to analysis.
Sediment	VOCs	Low	EPA Method 5035/8260B	En Core™ samplers	3 En Cores™ per sample plus a 4-oz. glass jar for moisture.	Preserve at the lab in sodium bisulfite	48 hours to preserve; 14 days from preservation to analysis
Sediment	SVOCs	Low	EPA Method 5030/8270C	At least 60 grams	1 -8 ounce glass jar	4°C	14 days to extraction; 40 days from extraction to analysis
Sediment	PCBs	Low	EPA Method 5030/8082	At least 60 grams	1 -8 ounce glass jar	4°C	14 days to extraction; 40 days from extraction to analysis
Sediment	Metals (except mercury)	Low	EPA Method 6010	1 gram	1 -8 ounce glass jar	4°C	180 days
Sediment	Mercury	Low	EPA Method 7471	1 gram	1 -8 ounce glass jar	4°C	28 days

[1] Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

QAPP Worksheet #20

(UFP-QAPP Manual Section 3.1.1)

Summarize by matrix, analytical group, and concentration level the number of field QC samples that will be collected and sent to the laboratory.

☐ Worksheet Not Applicable (State Reason)

Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Field Duplicate Pairs	Inorganic No. of MS	No. of Field Blanks	No. of Equip. Blanks	No. of PT Samples	Total No. of Samples to Lab
Groundwater	VOCs	Low		16	1	2	1	NA	NA	20
Groundwater	SVOCs	Low		16	1	2	1	NA	NA	20
Sediment	VOCs	Low		TBA	TBA	TBA	TBA	TBA	TBA	TBA
Sediment	SVOCs	Low		TBA	TBA	TBA	TBA	TBA	TBA	TBA
Sediment	PCBs	Low		TBA	TBA	TBA	TBA	TBA	TBA	TBA
Sediment	metals	Low		TBA	TBA	TBA	TBA	TBA	TBA	TBA

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

QAPP Worksheet #21

(UFP-QAPP Manual Section 3.1.2)

List all SOPs associated with project sampling including, but not limited to, sample collection, sample preservation, equipment cleaning and decontamination, equipment testing, inspection and maintenance, supply inspection and acceptance, and sample handling and custody. Include copies of the SOPs as attachments or reference all in the QAPP. Sequentially number sampling SOP references in the Reference Number column. The reference number can be used throughout the QAPP to refer to a specific SOP.

☐ Worksheet Not Applicable (State Reason)

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Check if yes)	Comments
NA	Sampling of Groundwater	URS Corp.	Low flow sampling	☐	Contained in the corresponding Sampling and Analysis Plan
NA	Sediment Sampling	URS Corp.		☐	Contained in the corresponding Sampling and Analysis Plan

QAPP Worksheet #22

(UFP-QAPP Manual Section 3.1.2.4)

Identify all field equipment and instruments (other than analytical instrumentation) that require calibration, maintenance, testing, or inspection and provide the SOP reference number for each type of equipment. In addition, document the frequency of activity, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Conductivity Meter	Calibration	NA	Battery	Visual	Daily	See Manufacturer's Manual	Adjust as needed	Field Team Leader	See corresponding Sampling and Analysis Plan
pH meter	Calibration	NA	Battery	Visual	Daily	See Manufacturer's Manual	Adjust as needed	Field Team Leader	See corresponding Sampling and Analysis Plan
Photoionization Detector	Calibration	NA	Battery	Visual	Daily	See Manufacturer's Manual	Adjust as needed	Field Team Leader	See corresponding Sampling and Analysis Plan

[1]Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

QAPP Worksheet #23

(UFP-QAPP Manual Section 3.2.1)

List all SOPs that will be used to perform on-site or off-site analysis. Indicate whether the procedure produces screening or definitive data. Sequentially number analytical SOP reference in the Reference Number column. Include copies of the SOPs as attachments or reference in the QAPP. The reference number can be used throughout the QAPP to refer to a specific SOP.

☐ Worksheet Not Applicable (State Reason)

Analytical SOP References Table

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
SOP-001-QAQC	Quality Assurance Project Plan-5/5/206	Definitive	All	All	FirstTechnology	No
SOP-OA-1000-VOCs	VOCs by GC/MS; 4/26/2006	Definitive	VOCs in Groundwater	GC/MS	FirstTechnology	☐
SOP-OA-1100-SVOCs	SVOCs by GC/MS; 4/24/2006	Definitive	SVOCs in Groundwater	GC/MS	FirstTechnology	☐
SOP-OE-1600-SVOC	Liquid Extraction of SVOCs in Water – 7/10/2006	Deifinitive	SVOCs in Groundwater	GC/MS	FirstTechnology	No
SOP-OE-1550-VOCs-Soil	VOCs in Soil by GC/MS-7/10/2006	Definitive	VOCs in Sediment	GC/MS	FirstTechnology	No
SOP-OE-1500-General Organic Extractions	General Organic Extractions – 7/10/2006	Definitive	General Extraction	NA	FirstTechnology	No
SOP-OE-1700-SVOCs	Extraction of SVOCs in Soil by Sonication – 10/25/2006	Definitive	SVOCs in Sediment	NA	FirstTechnology	No
SOP-OE-2000-PCBs	Extraction of PCBs in Soil by Sonication – 10/25/2006	Definitive	PCBs in Sediment	NA	FirstTechnology	No
SOP-OE-1950-PCB Cleanup	Clean Up of PCB Extracts – 10/11/2006	Definitive	PCBs in Sediment/Soils	NA	FirstTechnology	No
SOP-OA-1500-PCBs	PCBs by GC/ECD	Definitive	PCBs in water and sediment	GC/ECD	FirstTechnology	No
SOP-M-1000-metals	Preparation of Metals	Definitive	Metals	NA	FirstTechnology	No

Analytical SOP References Table

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
SOP-M-1500-metals	Metals by ICP	Definitive	Metals	ICP/Cold Vapor AA	FirstTechnology	No

QAPP Worksheet #24

(UFP-QAPP Manual Section 3.2.2)

Identify all analytical instrumentation that requires calibration and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/MS (VOCs)	According to SW-846 EPA Method 8260B; Initial Calibration (ICV); Continuing Calibration (CCV)	ICV=as needed. CCV=daily	ICV: %RSD =± 30% for all compounds. RRF>0.05 CCV: %D=±25%; RRF>0.05	ICV: rerun 5-point calibration curve. CCV: rerun CCV; check ICV. Rerun ICV if necessary.	GC/MS analyst; GC/MS supervisor	SOP-OA-1000-VOCs SOP-OE-1550-VOCs-Soil
GC/MS (SVOCs)	According to SW-846 EPA Method 8270C; Initial Calibration (ICV); Continuing Calibration (CCV)	ICV=as needed. CCV=daily	ICV: %RSD =± 30% for all compounds. RRF>0.05 CCV: %D=±25%; RRF>0.05	ICV: rerun 5-point calibration curve. CCV: rerun CCV; check ICV. Rerun ICV if necessary.	GC/MS analyst; GC/MS supervisor	SOP-OA-1100-SVOCs
GC/ECD	According to SW-846 EPA Method 8082A; Initial Calibration (ICV); Continuing Calibration (CCV)	ICV=as needed. CCV=daily	ICV=-50% - +100% peak area. RT window =).06 minutes CV=±20%	ICV: rerun 5-8 point calibration curve. CCV: rerun CCV; check ICV. Rerun ICV if necessary.	GC/MS analyst; GC/MS supervisor	SOP-OA-1500-PCBs

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
ICP	According to SW-846 EPA Method 6010B; Initial Calibration (ICV); Continuing Calibration (CCV)	ICV=beginning of batch of 20 samples. CCV=every 2 hours or 20 samples.	ICV=80-120% CCV=90-110%	ICV=If the initial calibration verification (including detection limit check, ICV, ICB, and RS) fails, repeat and/or make fresh standards as needed. Do not run any samples until acceptable results are obtained. CCV= Repeat and/or make fresh standards as needed. Adjust calibration or redo calibration. If calibration was adjusted or redone, when QC is acceptable, re-analyze all samples since the last acceptable continuing calibration verification.	Metals supervisor	SOP-M-1000-metals

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
Cold Vapor AA (mercury)	According to SW-846 EPA Method 7471A; Initial Calibration (ICV); Continuing Calibration (CCV)	ICV=beginning of batch of 20 samples. CCV=every 2 hours or 20 samples.	ICV=80-120% CCV=90-110%	ICV=If the initial calibration verification (including detection limit check, ICV, ICB, and RS) fails, repeat and/or make fresh standards as needed. Do not run any samples until acceptable results are obtained. CCV= Repeat and/or make fresh standards as needed. Adjust calibration or redo calibration. If calibration was adjusted or redone, when QC is acceptable, re-analyze all samples since the last acceptable continuing calibration verification.	Metals supervisor	SOP-M-1000-metals

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

QAPP Worksheet #25

(UFP-QAPP Manual Section 3.2.3)

Identify all analytical instruments that require maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
GC/MS	Recondition column. Change column. Clean MS sources	ICV, CCV, surrogate recoveries RRFs out of criteria. MS Tune out of criteria	Check chromatograms. Check connections. Check for leaks. Check MS tune	As needed. As needed.	Criteria from methods 8260, 8270 Criteria from methods 8260, 8270	Clean column. Change column. Take apart MS and clean source. Inspect system; correct problem; rerun calibration and affected samples.	GC/MS supervisor	SOP-OA-1100 VOC; SOP-OA1100-SVOC
GC	Recondition column. Change column.	ICV, CCV, surrogate recoveries RRFs out of criteria.	Check chromatograms. Check connections. Check for leaks.	As needed	Criteria from EPA Method 8082	Clean column. Change column. Inspect system; correct problem; rerun calibration and affected samples.	GC supervisor	SOP-OA1500-PCBs
ICP	Replace disposable lines. Flush lines.	NA	Check connections. Check for leaks.	As needed.	Criteria from EPA Method 6010	Inspect system; correct problem; rerun calibration and affected samples.	Metals supervisor	SOP-M-1000-metals
Cold Vapor AA	Replace disposable lines. Flush lines.	NA	Check connections. Check for leaks.	As needed.	Criteria from EPA Method 7471A.	Inspect system; correct problem; rerun calibration and affected samples.	Metals supervisor	SOP-M-1000-metals

[1]Specify the appropriate reference letter or number from Analytical SOP References table (Worksheet #23).

QAPP Worksheet #26

(UFP-QAPP Manual Appendix A)

Use this worksheet to identify components of the project-specific sample handling system. Record personnel, and their organizational affiliations, who are primarily responsible for ensuring proper handling, custody, and storage of field samples from the time of collection, to laboratory delivery, to final sample disposal. Indicate the number of days field samples and their extracts/digestates will be archived prior to disposal.

☐ Worksheet Not Applicable (State Reason)

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT	
Sample Collection (Personnel/Organization):	F ield Team/URS Corporation
Sample Packaging (Personnel/Organization):	F ield Team/URS Corporation
Coordination of Shipment (Personnel/Organization):	F ield Team/URS Corporation
Type of Shipment/Carrier:	Courier Service. Laboratory pick up.
SAMPLE RECEIPT AND ANALYSIS	
Sample Receipt (Personnel/Organization):	Shipping and Receiving/First Technology Inc.
Sample Custody and Storage (Personnel/Organization):	Sample Custodian/First Technology Inc.
Sample Preparation (Personnel/Organization):	Extactions Laboratory (organics)/metals preparation laboratory/First Technology Inc.
Sample Determinative Analysis (Personnel/Organization):	GC/MS laboratory analyst/GC analyst/metals analyst/First Technology Inc.
SAMPLE ARCHIVING	
Field Sample Storage (No. of days from sample collection):	45 days from date of sample collection.
Sample Extract/Digestate Storage (No. of days from extraction/digestion):	organics (SVOCs, PCBs) extractes= 60 days from extraction; metals digestates= 180 days from digestion.
Biological Sample Storage (No. of days from sample collection):	NA
SAMPLE DISPOSAL	
Personnel/Organization:	First Technolgy, Inc.
Number of Days from Analysis:	Organics (45 days past analyses); metals (6 months).

QAPP Worksheet #27

(UFP-QAPP Manual Section 3.3.3)

Describe the procedures that will be used to maintain sample custody and integrity. Include examples of chain-of-custody forms, traffic reports, sample identification, custody seals, laboratory sample receipt forms, and laboratory sample transfer forms. Attach or reference applicable SOPs.

☐ Worksheet Not Applicable (State Reason)

Sample Custody Requirements

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

Field personnel are responsible for the care and custody of samples from the time they are collected until they are transferred to the sample shipper or the laboratory. Field logbooks will provide the means of recording data collecting activities performed. As such, entries will be described in as much detail as possible so that persons going to the facility could reconstruct a particular situation without reliance on memory.

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

Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored with the project files when not in use. Each logbook will be identified by the project name and a unique project number.

All necessary sample bottles, preservatives, and reagents will be shipped to URS by the laboratory and received by URS field personnel. Sample bottles needed for a specific sampling task will be released by the field team leader to the sampling team after the integrity of the bottles has been verified. The sample bottle requirements for the samples to be collected during the investigation are provided above in QAPP worksheet #19.

Samples will be collected in accordance with the Sampling and Analysis Plan (SAP). Each sample will be assigned a unique sample identification number. Field duplicates will also be assigned a unique sample identification number. The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible will handle the samples. Samples will be labeled according to the project SAP.

Immediately after sample collection, the sample will be placed on ice in a rigid thermal shipping container and maintained at 4°C for shipment to the laboratory. The container will remain in the sampler's view or locked in the sampling vehicle for temporary storage. The sampler will fill out a chain of custody (COC) form that will account for each sample contained in each sampling container. Field blanks and other QC samples will be sent with each group of samples. To ensure the laboratory does not know the identity of the QC duplicate samples, the COC forms accompanying the samples will not identify the duplicate sample as such. The URS copy of the COC form will identify the QC samples and be retained as a permanent record in the project files.

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

The laboratory will assume custody of the samples on receipt. The laboratory Sample Manager will record the date and time and sign the COC form. The Sample Manager will immediately inspect the shipment for damage and completeness and will report any problems to the URS QA Officer or URS Project Manager. First Technology custody procedures including bottle preparation before shipment to the site, sample receiving and log-in, sample storage and numbering, measures taken to confirm proper sample preservation, sample security, tracking during sample preparation and analysis, and storage of data are described in the laboratory's QAPP (SOP-001-QAQC).

Sample Identification Procedures:

Groundwater samples will be identified by the monitoring well number. Sediment samples will be identified by field personnel in the field notebook. See QAPP Worksheet #18.

Chain-of-custody Procedures:

Field sampling personnel will complete the COC form. The sampler will record the date and time and sign in the appropriate block of the form to relinquish custody. The name of the courier that will be transporting the samples will be identified in an appropriate block of the chain-of-custody form along with the air-bill number. The original COC record will be placed inside the shipping container in a protective plastic bag. The shipping container will then be sealed using adhesive custody seals attached to the front and back of the cooler. The custody seals will be covered using clear plastic tape and the cooler will be strapped shut using strapping tape in at least two locations. The seals will be used to identify if the shipping container remained closed during shipment. The sample shipping receipt will be retained as part of the permanent COC document. Samples will be transported to the laboratory the same day that the samples are collected in the field using an overnight carrier. Field personnel will verify that all sample vials are labeled correctly and that COC records are properly completed before shipment to the laboratory.

QAPP Worksheet #28

(UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

QC Samples Table

Matrix	Groundwater
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Detrex Source Control Area Remedial Design/Remedial Action Operation, Maintenance and Monitoring Plan DNAPL Recovery System
Analytical Method/ SOP Reference	EPA 8260B/OA-1000-VOCs
Sampler's Name	TBA
Field Sampling Organization	URS Corporation
Analytical Organization	First Technology, Inc.
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria

QC Samples Table

Matrix	Groundwater
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Detrex Source Control Area Remedial Design/Remedial Action Operation, Maintenance and Monitoring Plan DNAPL Recovery System
Analytical Method/ SOP Reference	EPA 8260B/OA-1000-VOCs
Sampler's Name	TBA
Field Sampling Organization	URS Corporation
Analytical Organization	First Technology, Inc.
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	1 per 20	See EPA 8260B	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples
Field duplicate	1 per 20	EPA 8260B – RPD=±30%	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples

QC Samples Table

Matrix	Groundwater
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Detrex Source Control Area Remedial Design/Remedial Action Operation, Maintenance and Monitoring Plan DNAPL Recovery System
Analytical Method/ SOP Reference	EPA 8260B/OA-1000-VOCs
Sampler's Name	TBA
Field Sampling Organization	URS Corporation
Analytical Organization	First Technology, Inc.
No. of Sample Locations	20

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 blank	1 per 20	EPA 8260B – no target compounds above RL	Qualify data as undetected if blanks contain target VOCs (use 5X/10X rule)	URS QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified

QC Samples Table

Matrix	Groundwater
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Detrex Source Control Area Remedial Design/Remedial Action Operation, Maintenance and Monitoring Plan DNAPL Recovery System
Analytical Method/ SOP Reference	EPA 8260B/OA-1000-VOCs
Sampler's Name	TBA
Field Sampling Organization	URS Corporation
Analytical Organization	First Technology, Inc.
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Trip blank	1 per 20	EPA 8260B	Qualify data as undetected if blanks contain target VOCs (use 5X/10X rule)	URS QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Laboratory control sample	1 per 20	EPA 8260B	Rerun samples	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified

QC Samples Table

Matrix	Groundwater
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Detrex Source Control Area Remedial Design/Remedial Action Operation, Maintenance and Monitoring Plan DNAPL Recovery System
Analytical Method/ SOP Reference	EPA 8260B/OA-1000-VOCs
Sampler's Name	TBA
Field Sampling Organization	URS Corporation
Analytical Organization	First Technology, Inc.
No. of Sample Locations	20

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 Method Blank	1 per 20	EPA 8260B	Qualify data as undetected if blanks contain target VOCs (use 5X/10X rule)	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Surrogate Recoveries	All samples	EPA 8260B	Rerun samples	Laboratory QA Officer		Data may be qualified

QAPP Worksheet #28

(UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

QC Samples Table

Matrix	Groundwater
Analytical Group	SVOCs
Concentration Level	Low
Sampling SOP	Detrex Source Control Area Remedial Design/Remedial Action Operation, Maintenance and Monitoring Plan DNAPL Recovery System
Analytical Method/ SOP Reference	EPA 8270B/OA-1100-SVOCs
Sampler's Name	TBA
Field Sampling Organization	URS Corporation
Analytical Organization	First Technology, Inc.
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	1 per 20	See EPA 8270C	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples
Field duplicate	1 per 20	EPA 8270C – RPD=±30%	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples
Field blank	1 per 20	EPA 8270C – no target compounds above RL	Qualify data as undetected if blanks contain target VOCs (use 5X/10X rule)	URS QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Laboratory control sample	1 per 20	EPA 8270C	Rerun samples	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Laboratory Method Blank	1 per 20	EPA 8270C	Qualify data as undetected if blanks contain target SVOCs (use 5X/10X rule)	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Surrogate Recoveries	All samples	EPA 8270C	Rerun samples	Laboratory QA Officer		Data may be qualified

QAPP Worksheet #28

(UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

QC Samples Table

Matrix	Sediment/Soil
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Detrex Source Control Area Remedial Design/Remedial Action Operation, Maintenance and Monitoring Plan DNAPL Recovery System
Analytical Method/ SOP Reference	EPA 5035/8260B/OA-1000-VOCs/SOP-OE-1550-VOCsSoil
Sampler's Name	TBA
Field Sampling Organization	URS Corporation
Analytical Organization	First Technology, Inc.
No. of Sample Locations	TBA

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	1 per 20	See EPA 8260B	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples
Field duplicate	1 per 20	EPA 8260B – RPD=±50%	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples
Field blank	1 per 20	EPA 8260B – no target compounds above RL	Qualify data as undetected if blanks contain target VOCs (use 5X/10X rule)	URS QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Laboratory control sample	1 per 20	EPA 8260B	Rerun samples	Laboratory QA Officer	see QAPP Worksheets #12 and #37.NA	Data may be qualified
Laboratory Method Blank	1 per 20	EPA 8260B	Qualify data as undetected if blanks contain target VOCs (use 5X/10X rule)	Laboratory QA Officer	see QAPP Worksheets #12 and #37.NA	Data may be qualified
Surrogate Recoveries	All samples	EPA 8260B	Rerun samples	Laboratory QA Officer		Data may be qualified

QAPP Worksheet #28

(UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

QC Samples Table

Matrix	Sediment/Soil
Analytical Group	SVOCs
Concentration Level	Low
Sampling SOP	Detrex Source Control Area Remedial Design/Remedial Action Operation, Maintenance and Monitoring Plan DNAPL Recovery System
Analytical Method/ SOP Reference	EPA 8270B/OA-1100-SVOCs/SOP-OE-1700-SVOC
Sampler's Name	TBA
Field Sampling Organization	URS Corporation
Analytical Organization	First Technology, Inc.
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	1 per 20	See EPA 8270C	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples
Field duplicate	1 per 20	EPA 8270C – RPD=±50%	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples
Field blank	1 per 20	EPA 8270C – no target compounds above RL	Qualify data as undetected if blanks contain target SVOCs (use 5X/10X rule)	URS QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Laboratory control sample	1 per 20	EPA 8270C	Rerun samples	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Laboratory Method Blank	1 per 20	EPA 8270C	Qualify data as undetected if blanks contain target SVOCs (use 5X/10X rule)	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Surrogate Recoveries	All samples	EPA 8270C	Rerun samples	Laboratory QA Officer		Data may be qualified

QAPP Worksheet #28

(UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

QC Samples Table

Matrix	Sediment/Soil
Analytical Group	PCBs
Concentration Level	Low
Sampling SOP	Detrex Source Control Area Remedial Design/Remedial Action Operation, Maintenance and Monitoring Plan DNAPL Recovery System
Analytical Method/ SOP Reference	EPA 8082/OE-2000-PCBs/OA-1500-PCBs/OA-1950-PCBCleanup
Sampler's Name	TBA
Field Sampling Organization	URS Corporation
Analytical Organization	First Technology, Inc.
No. of Sample Locations	TBA

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	1 per 20	See EPA 8082	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples
Field duplicate	1 per 20	EPA 8082 – RPD=±50%	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples
Field blank	1 per 20	EPA 8082 – no target compounds above RL	Qualify data as undetected if blanks contain target PCBs (use 5X rule)	URS QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Laboratory control sample	1 per 20	EPA 8082; R=70-130	Rerun samples	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Laboratory Method Blank	1 per 20	EPA 8082	Qualify data as undetected if blanks contain target PCBs (use 5X rule)	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Surrogate Recoveries	All samples	EPA 8082	Rerun samples	Laboratory QA Officer		Data may be qualified

QAPP Worksheet #28

(UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

QC Samples Table

Matrix	Sediment/Soil
Analytical Group	Metals
Concentration Level	Low
Sampling SOP	Detrex Source Control Area Remedial Design/Remedial Action Operation, Maintenance and Monitoring Plan DNAPL Recovery System
Analytical Method/ SOP Reference	EPA 6010/7471
Sampler's Name	TBA
Field Sampling Organization	URS Corporation
Analytical Organization	First Technology, Inc.
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	1 per 20	See EPA 6010/7471; limits=75-125;Hg= 80-110	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples
Field duplicate	1 per 20	EPA 6010/7471 – RPD=±50%	Use Professional judgement in qualifying data.	URS QA Officer	see QAPP Worksheets #12 and #37.	Dependent on other field and laboratory QC samples
Field blank	1 per 20	EPA 6010/7471 – no target analyte above RL	Qualify data as undetected if blanks contain target analytes(use 5X)	URS QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Laboratory control sample	1 per 20	EPA 6010/7471; R=80-120	Rerun samples	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Laboratory Method Blanks (ICB, CCB, prep. Blank)	1 per 20	EPA 6010/7471	Qualify data as undetected if blanks contain target analyte (use 5Xrule)	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Interference check	1 per 20	EPA 6010/7471; R=80-120	Rerun samples	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified
Serial dilution	1 per 10	EPA 6010/7471; %D≤10	Rerun samples	Laboratory QA Officer	see QAPP Worksheets #12 and #37.	Data may be qualified

QAPP Worksheet #29

(UFP-QAPP Manual Section 3.5.1)

Identify the documents and records that will be generated for all aspects of the project including, but not limited to, sample collection and field measurement, on-site and off-site analysis, and data assessment.

☐ Worksheet Not Applicable (State Reason)

Project Documents and Records Table

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Notes	Sample Receipt, Custody, and Tracking Records	Sample Receipt, Custody, and Tracking Records	Field Audit Checklist	
Chain of Custody	Standard tracable logs	Standard tracable logs	Laboratory Audit Checklist	
Custody Seals	Sample Preparation/Extraction Logs	Sample Preparation/Extraction Logs	Data Validation/Usability Review	
Corrective Action Forms	Run Logs	Run Logs	Corrective Action Reports	
	Equipment Maintenance, Testing and Inspection Logs	Equipment Maintenance, Testing and Inspection Logs		
	Corrective Action Forms	Corrective Action Forms		
	Reported Field Sample Results	Reported Field Sample Results		
	Sample Disposal Records	Reported Results for standards, QC checks, and QC samples		
		Data Package Completenese Checklist		
		Sample Disposal Records		

Project Documents and Records Table

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
		Extraction/Clean up records		
		Raw Data (as pdf on CD)		

QAPP Worksheet #30

(UFP-QAPP Manual Section 3.5.2.3)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify all laboratories or organizations that will provide analytical services for the project, including on-site screening, on-site definitive, and off-site laboratory analytical work. If applicable, identify the subcontractor laboratories and backup laboratory or organization that will be used if the primary laboratory or organizations cannot be used.

☐ Worksheet Not Applicable (State Reason)

Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number
Water	VOCs	Low	NA	SOP-OA-1000-VOCs	30 days from receipt of samples	First Technology, Inc. 4450 Johnston Parkway Unit B Cleveland, OH 44128 Contact: Phil Byron (216) 663-0808	STL Inc. 4101 Shuffel Drive NW North Canton, OH 44720 Contact: Mark Loeb (330) 497-9396
Water	SVOCs	Low	NA	SOP-OA-1100-SVOCs	30 days from receipt of samples	First Technology, Inc. 4450 Johnston Parkway Unit B Cleveland, OH 44128 Contact: Phil Byron (216) 663-0808	STL Inc. 4101 Shuffel Drive NW North Canton, OH 44720 Contact: Mark Loeb (330) 497-9396
Sediment	VOCs	Low	NA	SOP-OA-1000-VOCs	30 days from receipt of samples	First Technology, Inc. 4450 Johnston Parkway Unit B Cleveland, OH 44128 Contact: Phil Byron (216) 663-0808	STL Inc. 4101 Shuffel Drive NW North Canton, OH 44720 Contact: Mark Loeb (330) 497-9396
Sediment	SVOCs	Low	NA	SOP-OA-1100-SVOCs	30 days from receipt of samples	First Technology, Inc. 4450 Johnston Parkway Unit B Cleveland, OH 44128 Contact: Phil Byron (216) 663-0808	STL Inc. 4101 Shuffel Drive NW North Canton, OH 44720 Contact: Mark Loeb (330) 497-9396
Sediment	PCBs	Low	NA	SOP-OA-1500-PCBs	30 days from receipt of samples	First Technology, Inc. 4450 Johnston Parkway Unit B Cleveland, OH 44128 Contact: Phil Byron (216) 663-0808	STL Inc. 4101 Shuffel Drive NW North Canton, OH 44720 Contact: Mark Loeb (330) 497-9396

Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number
Sediment	metals	Low	NA	SOP-M-1000-metals	30 days from receipt of samples	First Technology, Inc. 4450 Johnston Parkway Unit B Cleveland, OH 44128 Contact: Phil Byron (216) 663-0808	STL Inc. 4101 Shuffel Drive NW North Canton, OH 44720 Contact: Mark Loeb (330) 497-9396

QAPP Worksheet #31
(UFP-QAPP Manual Section 4.1.1)
Identify the type, frequency, and responsible parties of planned assessment activities that will be preformed for the project.
Field and Laboratory audits are not planned during this phase of the project unless requested by EPA.

Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Project safety analysis	start of project	internal	URS	Health and Safety Officer	Health and Safety Officer	Field Team Leader	Project Manager

QAPP Worksheet #32

(UFP-QAPP Manual Section 4.1.2)

For each type of assessment describe procedures for handling QAPP and project deviations encountered during the planned project assessments.

☐ Worksheet Not Applicable (State Reason)

Field and Laboratory audits are not planned during this phase of the project unless requested by EPA.

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, Org.)	Timeframe for Response
Health and Safety	Safe work practices	Health and Safety officer	Immediate	Correct behavior	Field Team Leader, URS	Immediate
NA	NA	NA	NA	NA	NA	NA

Project Manag

QAPP Worksheet #33

(UFP-QAPP Manual Section 4.2)

Identify the frequency and type of planned QA Management Reports, the projected delivery date, the personnel responsible for report preparation, and the report recipients.

☐ Worksheet Not Applicable (State Reason)

QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Groundwater Monitoring Report/Data Usability Summary	Quarterly	Ongoing	David Blaushild, Project Chemist/Peggy Schuler, QA Officer/Martin L. Schmidt, Project Manager; URS Corp.	Tom Steib, Plant Manager; Detrex Corp.; Tom Doll, Operations Manager, Detrex Corp.; Terese Van Donsel, Remedial Project Manager, USEPA Region 5.
Trench Groundwater Sampling Data Usability Summary	One time event	June 2007	David Blaushild, Project Chemist/Peggy Schuler, QA Officer/Martin L. Schmidt, Project Manager; URS Corp.	Tom Steib, Plant Manager; Detrex Corp.; Tom Doll, Operations Manager, Detrex Corp.; Terese Van Donsel, Remedial Project Manager, USEPA Region 5.
DS Sediment Sampling Data Usability Summary	One time event	Summer 2007	David Blaushild, Project Chemist/Peggy Schuler, QA Officer/Martin L. Schmidt, Project Manager; URS Corp.	Tom Steib, Plant Manager; Detrex Corp.; Tom Doll, Operations Manager, Detrex Corp.; Terese Van Donsel, Remedial Project Manager, USEPA Region 5.

QAPP Worksheet #34

(UFP-QAPP Manual Section 5.2.1)

Describe the processes that will be followed to verify project data. Verification inputs include items such as those listed in Table 9 of the UFP-QAPP Manual (Section 5.1). Describe how each item will be verified, when the activity will occur, and what documentation is necessary, and identify the persons responsible. *Internal* or *external* is in relation to the data generator.

☐ Worksheet Not Applicable (State Reason)

Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain of Custody and sample delivery forms	Chain of Custody (COC) forms and sample delivery documentation will be sent with the physical samples to the laboratory. A copy of the COC will be retained by the field personnel and placed in the project file. The field personnel packing the sample coolers will sign the COC. Sample Receiving at the laboratory will sign the COC upon sample receipt. A copy of the COC will be redtained at the laboratory. A copy of the COC will be submitted with the data deliverables.	I	Field Team Leader, URS Corp. Scott Bolam, First Technology, Inc.
Field Notes	Field notes will be reviewed internally and placed in the project file.	I	Martin L. Schmidt, URS Corp.
Laboratory Data	All laboratory data deliverables will be verified internally by the laboratory QA Officer for completeness and technical accuracy prior to submittal. All received data packages will be verified using documents mentioned in QAPP Worksheet #14.	I E	Scott Bolam, First Technology, Inc. Peggy Schuler, URS Corp.

QAPP Worksheet #35

(UFP-QAPP Manual Section 5.2.2)

Describe the processes that will be followed to validate project data. Validation inputs include items such as those listed in Table 9 of the UFP-QAPP Manual (Section 5.1). Describe how each item will be validated, when the activity will occur, and what documentation is necessary and identify the person responsible. Differentiate between steps IIa and IIb of validation.

☐ Worksheet Not Applicable (State Reason)

Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIb	Onsite Analyses (field measurements only)	All onsite field data will be reviewed against QAPP requirements for completeness and accuracy.; verified against field notes.	Peggy Schuler/David Blaushild, URS Corp.
IIa	SOPs	Ensure that all sampling and analytical SOPs are followed.	Peggy Schuler/David Blaushild, URS Corp.
IIa	Documentation of Method QC Results	Establish that all method required QC samples were analyzed and met required limits.	Peggy Schuler/David Blaushild, URS Corp.
IIb	Documentation of Method QC Results	Establish that all QAPP required QC samples were analyzed and met required limits.	Peggy Schuler/David Blaushild, URS Corp.
IIb	Project Quantitation Limits	All samples met project quantitation limits.	Peggy Schuler/David Blaushild, URS Corp.
IIa	Analytical data	Review of analytical data.	Peggy Schuler/David Blaushild, URS Corp.

QAPP Worksheet #36

(UFP-QAPP Manual Section 5.2.2)

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

☐ Worksheet Not Applicable (State Reason)

Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa	Groundwater	VOCs	Low	See QAPP Worksheet #14 under Data Review Tasks and QAPP Worksheet #28..	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.
IIa	Groundwater	SVOCs	Low	See QAPP Worksheet #14 under Data Review Tasks and QAPP Worksheet #28..	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.
IIa	Sediment	VOCs	Low	See QAPP Worksheet #14 under Data Review Tasks and QAPP Worksheet #28..	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.
IIa	Sediment	SVOCs	Low	See QAPP Worksheet #14 under Data Review Tasks and QAPP Worksheet #28..	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.
IIa	Sediment	PCBs	Low	See QAPP Worksheet #14 under Data Review Tasks and QAPP Worksheet #28..	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.
IIa	Sediment	Metals	Low	See QAPP Worksheet #14 under Data Review Tasks and QAPP Worksheet #28..	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.
IIb	Groundwater	VOCs	Low	QAPP Worksheets 12, 15, and 24.	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.

Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIb	Groundwater	SVOCs	Low	QAPP Worksheets 12, 15, and 24.	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.
IIb	Sediment	VOCs	Low	QAPP Worksheets 12, 15, and 24.	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.
IIb	Sediment	SVOCs	Low	QAPP Worksheets 12, 15, and 24.	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.
IIb	Sediment	PCBs	Low	QAPP Worksheets 12, 15, and 24.	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.
IIb	Sediment	Metals	Low	QAPP Worksheets 12, 15, and 24.	Peggy Schuler, Environmental Scientist or David Blaushild, Project Chemist; URS Corp.

QAPP Worksheet #37

(UFP-QAPP Manual Section 5.2.3)

Describe the procedures/methods/activities that will be used to determine whether data are of the right type, quality, and quantity to support environmental decision-making for the project. Describe how data quality issues will be addressed and how limitations of the use of the data will be handled.

☐ Worksheet Not Applicable (State Reason)

Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:
The Data Usability will be perfomed by personnel from URS Corp. Martin Schmidt, Project Manager will be resoponsibe for information in the Usability Assessment. He will also be responsible for assigning task work to the individual task members who will be supporting the Data Usability Assessment. Af ter the Data Usability Assessment, data deamed usable for project purposes will be used to evaluate impacts from chemicals of concern to groundwater and sediment at the site. The following items will be assessed in the data usability.

Sensitivity

The sensitivity required for each analyte measured in groundwater and sediment is based on standard method specified quantitation limits included URS EPA SW-846. The analytes to be measured during this project are presented on Worksheet #15. Worksheet #15 includes project quantitation limits and method detection limits for the laboratory.

Most quantitation limits (QLs) are designed to meet standard screening standards. QLs for sediments are dependent on moisture content of the sample. QLs for sediments presented on Worksheet #15 assume that the sample is 100 percent solid. QLs for sediment in the data will be reported on a dry weight basis and will be higher than those stated in Worksheet #15. Groundwater QLs are dependent on sample size available for extraction, preparation and analysis. In the rare instance where a lower than method specified volume of groundwater is available, elevated QLs will be reported. In the rare instance that an analyte has a clean up standard for sediment or groundwater that are lower than the method specified EQL, the laboratory will report to the method detection limit (MDL). MDLs represent the lowest theoretical detection limit for a given analyte that can be achieved with any confidence. MDLs are determined by the laboratory on an annual basis using statistical methods in accordance with 40CFR136. As such, the MDLs can change from year to year. In rare cases where the clean up standard is son low that no known or accepted analytical method exists that will meet these criteria, (some VOCs and PAHs in groundwater fall into this category) the laboratory will default to the MDL.

Precision

Definition

Precision is defined as the degree to which two or more measurements are in agreement. It is expressed as relative percent difference (RPD). The calculation for precision is presented below.

Field QC Precision Objectives

Field precision is assessed through the collection of field duplicate samples and the use of MS/MSD samples. Field duplicates and MS/MSD will be collected at the rate of 1 per 20 samples per matrix. Criteria to measure field duplicates and MS/MSD are presented in Worksheet #12.

Laboratory QC Precision Objectives

Laboratory precision is assessed through the use of laboratory duplicates and LCS/LCSD samples. Laboratory and method specific precision is presented in the laboratory's QAPP, which is included with this QAPP as a pdf on CD.

Precision Assessment

Precision is assessed through the analysis of MS/MSD, duplicate LCS, laboratory duplicate (inorganics) and field duplicate analysis. Precision is expressed as relative percent difference (RPD). Precision is calculated as follows:

$$RPD = \frac{S - D}{\frac{(S+D)}{2}} \times 100$$

Where: S = First sample value (e.g., original MS value)
D = Second sample value (e.g., duplicate MSD value)

Accuracy

Definition

Accuracy is defined as the degree to which analytical measurements agree with a reference value. The calculation for accuracy is presented below.

Field Accuracy Objectives

Field accuracy is assessed through the use of trip and equipment blanks. Equipment and trip blanks will be collected at the rate of 1 per 20 samples. Trip blanks will be collected and analyzed for VOCs only. Assessment criteria for blanks are presented in Worksheet #12.

Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the measurement of MS recoveries, LCS recoveries, and surrogate recoveries. Percent recoveries will be assessed against established method and laboratory criteria. Laboratory criteria are presented in the laboraory's QAPP accompanying this document.

Accuracy Assessment

Accuracy is assessed through the analysis of blank spikes, matrix spikes, surrogates, and other laboratory control samples. Sample spikes are included in every set of 20 samples tested on each instrument. Accuracy is expressed through percent recovery (%R). Accuracy is calculated as follows:

$$\textit{PERCENT RECOVERY} = \frac{(C_2 - C_1)}{C_0} \times 100\ \%$$

Where: C_2 = measured value of spiked sample,
C_1 = measured value of the unspiked value,
C_0 = known amount of the spike in the sample.

Completeness

Definition

Completeness is a qualitative measure of the amount of valid data obtained from a measurement program compared to the amount that was expected to be obtained under normal conditions. The equation for completeness is presented below.

Field Completeness Objectives

Field completeness is a measure of the number of samples collected and analyzed versus the number of samples proposed in the Workplan. Field completeness is expected to be 90 percent or greater.

Laboratory Completeness Objectives

Laboratory completeness is a measure of the number of measurements that meet project DQOs versus the number of samples analyzed. Laboratory completeness is expected to be 90 percent or greater.

Completeness Assessment

Completeness is assessed through the comparison of the total number of samples analyzed with the total number of valid sample results. Completeness is expressed as a ratio. Completeness is calculated as follows:

$$\text{COMPLETENESS} = \frac{(\text{ number of valid measurements })}{(\text{number of measurements planned})}$$

Representativeness

Definition

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Field Representativeness Objectives

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the Work plan is followed and that the proper sampling techniques are used.

Laboratory Representativeness Objectives

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting required holding times, and analyzing and assessing field duplicate samples. The sampling network was designed to provide data representative of facility conditions. The analytical results must adequately characterize the media that were being investigated.

Comparability

Definition

Comparability is an expression of the confidence with which one data set can be compared with another.

Field Comparability Objectives

Field comparability is dependent upon the proper use of sampling techniques and will be satisfied by ensuring that the Work plan is followed.

Laboratory Comparability Objectives

The laboratory objective for comparability is to strive toward the comparability of sample parameters on similar matrices as they relate to precision and accuracy determinations. Strict adherence to QA/QC procedures promotes the comparability of one set of reference data to another or comparability of data among all sample points. Analytical data comparability is achieved by following the sampling and analytical methods presented in the QAPP.

Describe the evaluative procedures used to assess overall measurement error associated with the project:
Comparative data from past investigations will be used to evaluate overall measurement. Field sampling measurements and laboratory measurement will be compared to accepted criteria.

Identify the personnel responsible for performing the usability assessment: The URS Corp. QA Officer and Project Chemist are responsible for performing the data usability assessment.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: The data usability assessment will be generated and presented in the sampling summary reports. Data will be tabulated and presented in the progress reports.

Resumes

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Martin L. Schmidt, Ph.D.

Director of Environmental Remediation

Areas of Expertise

Environmental Remediation
RCRA & CERCLA Assessment
Ohio VAP

Years of Experience

With URS: 22 Years
With Other Firms: 7 Years

Education

Ph.D./Engineering Geology/1985/Kent State University
MS/Soil Science/1977/Ohio State University
BS/Conservation/1975/Kent State University

Registration/Certification

Ohio Certified Professional VAP, No. 170
Certified Hazardous Materials Manager, No. 10881
Certified Professional Geologist, American Institute of Professional Geologists

Overview

Dr. Schmidt has more than 29 years of experience in environmental, hazardous waste and remediation projects. He has been responsible for work plan preparation, coordination of field activities, report preparation, project management, financial management and client relations on a variety of RCRA and CERCLA assessment and remediation projects. He has been involved with petroleum remedial activities at several refineries in Ohio and Kentucky and more than 200 UST sites throughout the United States involving soil and groundwater cleanup.

Since 1995 Dr. Schmidt has been involved with the Ohio Voluntary Action Program. During the rulemaking period he provided technical input regarding groundwater classification rule making. He has been a Certified Professional for the past 8 years. He has submitted six No Further Action letters and has received three Covenant-Not-Sue submittals from Ohio EPA on remedial site.

Dr. Schmidt is currently serving as the Director of Environmental Remediation the URS Cleveland, Ohio office. He is responsible for managing and coordinating environmental projects completed by a staff of 15 environmental professional and technical personnel.

Clients serviced by this group include several northern Ohio Fortune 500 industrial and commercial corporations, local municipalities, and law firms. During the past 22 years with URS, he has managed or coordinated more than 500 environmental projects in Ohio, throughout the U.S. and overseas including the following:

- RCRA investigations, assessments, closures, and corrective measures
- CERCLA investigations, assessments, remedial designs and remedial action
- EE/CA investigations, assessment, designs and removal action
- Phase I Assessments
- Phase II Assessments
- Environmental Compliance and HSE Audits
- NPDES, Air and Part B Permit Applications;
- Ohio Voluntary Action Program projects
- Asbestos assessment and abatement projects
- UST assessments, closures, remedial designs and remediation
- Environmental Liability Analysis
- Environmental Strategic Planning
- Remedial Negotiations with regulatory agencies

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In addition to consulting experience, Dr. Schmidt has taught environmental and geological science courses at Cleveland State University and John Carroll University as an Adjunct Professor.

Project Specific Experience

BP Amoco, Rockefeller No. 1 and No. 2 Refineries, Cleveland, Ohio (1995-2004): Currently serving as Certified Professional for voluntary remediation of two former refinery properties located in the industrial flats. Project oversight has included Phase I, Phase II, Remedial Design, Construction Quality Assurance, NFA preparation, Long-Term Operation / Management and Risk Mitigation Plans. Both sites encompass approximately 50 acres and have been impacted by LNAPL and fuel related compounds in the subsurface soil. Remedial activities at both properties have included the following components: sheetpile cutoff walls, protective soil cover, recovery sumps and redesign of stormwater drainage. A Covenant-Not-to-Sue from Ohio EPA was received for both properties in 2004. Commercial site development is anticipated following approval at both properties.

BP Amoco, Broadway Technical Center, Cleveland, Ohio (1995-2002): Currently serving as Certified Professional for site redevelopment of an automotive testing and research fuel blending facility. Project oversight has included Phase I, Phase II, Risk Assessment and NFA preparation. Site remedial activities have included demolition of facility buildings, removal of underground storage / above ground storage tanks, site-wide drainage controls, groundwater modeling, and LNAPL recovery.

BP Oil, Toledo Refinery, Toledo, Ohio (1995-present):
Currently serving as Refinery Alliance Manager for all environmental and remediation support at the 600-acre Toledo Refinery. Since 1995 a variety of U.S. EPA and Ohio EPA RCRA projects have been completed and include the following:

- Phase I RFI Work Plan, QAPP, and Field Sampling Plans;
- Phase I RFI Report, Human Health Risk Assessment and Ecological Risk Assessment;
- Risk-Based Closure Plans (Land Treatment Units, Jake's Lake, Main Pond, and Auxiliary Pond); and
- In-place Sludge Stabilization Closure Plan and Design (North Pond).

In addition to these remedial and closure activities, project work has included long-term liability analysis to manage environmental reserves, waste and water permitting services and review of site hydrogeology and sewer components.

BP Oil, Lima Refinery, Lima, Ohio (1995-present): Currently serving a Refinery Alliance Manager for all environmental and remediation support at the 600-acre Lima Refinery. Since 1995 a variety of U.S. EPA and Ohio EPA RCRA projects have been completed and include the

following:

- Phase I RFI Work Plan, QAPP, and Field Sampling Plans;
- Phase I RFI Report, Human Health Risk Assessment and Ecological Risk Assessment;
- Phase II RFI Work Plan;
- Phase II RFI Report;
- Corrective Measures Study;
- Risk-Based Closure Plans (Land Treatment Units,);
- In-place Stabilization Closure Plan (C-Pond, AFU Oily Sludge Pond); and
- Voluntary Corrective Action Plans (E-Pond, Land Treatment Unit);

In 1998 BP Oil sold the Lima Refinery to Premcor. Since the sale, on-site construction management and field engineering design support has been provided for annual budget preparation, storm water redesigns, sewer integrity surveys, LNAPL recovery operations, landfill closure monitoring and groundwater monitoring.

BP Oil, Lima Refinery "C" Pond Closure, Lima, Ohio (2001): Currently serving a Project Manager for the closure of two hazardous waste sludge storage ponds formerly used to store refinery wastewater sludge. The closure project is being coordinated with Ohio EPA and involves landfill closure of a 3-acre pond and clean closure of a 5-acre pond. The dirty closure component involves stabilization of 30,000 cubic yards of sludge with Portland cement. Treatability study tests provided a design mix of 25% cement to achieve 20 psi and non-leaching. The stabilized sludge was placed under a RCRA multi-layer cap. The project was completed in December 2001.

BP Oil, Cleveland, Ohio (1991-1999, 2005-present): Currently serving as Program Director for BP Oil Midwest Business Unit projects in Ohio. During the past 16 years more than 200 UST projects have been completed. Project work has included a variety of underground storage tank services including: site assessments, RDCA risk assessments, corrective action plan preparation, remedial system installation and operation and maintenance oversight. Active remediation projects include: groundwater pump and treatment, in situ bioremediation, dual phase and soil vapor extraction.

BP Pipeline, Canton, Ohio (1995-2002): Served as Project Manager and Project Director for a remedial design project dealing with a 2-million gallon gasoline release in a residential housing development. Project work has included design investigation sampling for soil vapor extraction and dual phase remediation technologies, developing site-specific risk based remediation goals, remedial system design and installation of six remedial systems. Currently, all operation and maintenance, NPDES permitting and reporting are being completed for the groundwater remedial systems that have been installed on the residential properties.

Ashland Inc., Catlettsburg Refinery, Ashland, Kentucky (1997-2001): Served as Project Manager for RCRA closure at the Catlettsburg Refinery, Route 3 Landfill. Project activities have included closure plan negotiations, landfill design, construction management, QA/QC, and closure certification. Additional responsibilities included annual groundwater monitoring, NPDES sampling, wastewater treatment plant, design and operation and coordination of removal of 5,000 cubic yards of sediment from a wastewater unit. As a result of using innovative geotube sediment dewatering techniques, $1.5 million in lost revenue was saved on the project.

Fields Brook Superfund Site, Ashtabula, Ohio (1992-1997): Served as Senior Project Manager for the Fields Brook Superfund Site, a CERCLA RI/FS and RD/RA project in northeast Ohio. The project included investigations of a Source Control Operable Unit, Sediment Operable Unit and Floodplain/Wetland Unit. The project area encompassed more than 5.6 square miles and has 17 industrial/manufacturing facilities. Results from the assessment and design investigations have been used to prepare design investigation work plans and to address remedial alternatives involving sediment solidification, incineration and landfilling of sediments containing PCBs and organics. Human and Ecological Risk assessments, Data Quality Objectives and statistical data evaluation approaches have been used to develop a cost effective remedial approach that involves stream channel excavation and wetland area remediation. In addition to site investigation work, project involvement included coordination with the final design team and negotiation strategies with U.S. EPA, Ohio EPA, and Natural Resource Trustees. Planned remediation will involve construction of an on-site landfill for more than 60,000 yds of sediment.

GenCorp, Point Pleasant West Virginia (1995-present): Serving as Project Coordinator for two Administrative Orders by Consent with U.S. EPA Region III. Project activities at the 80-acre former PVC manufacturing plant have included Consent Order negotiations, EE/CA Work Plan preparation, Response Action Plan development, design of GAC treatment system for municipal water supply impacted by chlorinated solvents and EECA investigations.

GenCorp, Akron, Ohio (1998-2000): Served as Project Manager for GenCorp's site acquisition program in eastern United States and worldwide. Project work has included air permitting, due diligence review for more than 50 sites being considered during facility expansions. Work scope involved coordinating overseas facility visits, Phase I, Phase II report preparation and development of cost projections.

Detrex Corporation, Ashtabula, Ohio (1995-present): Currently serving as Project Manager on a multi-task remediation program under Consent Order with USEPA Region V. This facility is part of the fields Brook Superfund Site. As part of the Source control Operable Unit, Detrex Corporation was required to mitigate DNAPL migration at this

Site. Project work has been ongoing since 1992 and includes the following: Consent Order Negotiations, site characterization, slurry wall design, groundwater recovery system design, DNAPL recovery design, construction management and remedial measures operation and maintenance.

Conrail Corporation, Ashtabula, Ohio (1995-present): Served as Project Coordinator for a CERCLA Remedial Design/Remedial Action project involving soil excavation and capping of a rail yard spill area. Site remediation involved excavation of 1,000 tons of soil and sediments, and was completed in 1998.

Diebold Corporation, North Canton, Ohio (1995-present): Served as Project Manager for Diebold Corporations site acquisition process. Project work has included Phase I and Phase II property assessment in the New York, Ohio, Australia and China, France, Germany and Brazil. Coordinated environmental liability analysis, compliance audits and installation of groundwater remedial systems for chlorinated solvents at two active facilities in Ohio and New York.

North Sewer Source Area (1997-2001): Served as PRP Group Project Coordinator for a CERCLA Remedial Design/Remedial Action project involving remediation of storm sewers containing contaminated sediment. The project work has included quantification and characterization of sediment within facility sewers, off-site sewers and adjacent stream channels and development of remedial alternatives. RD/RA Work Plans have been prepared to describe construction specifications and execution of a sediment containment remedial alternative. Remedial activities included sewer replacement and grouting of more than 2,000 feet of sewer. Remedial work was completed in Spring 2001.

Stickney-Tyler Landfill, Toledo, Ohio (1994-1995): Served as Field Investigation Task Manager for EE/CA (Engineering Evaluation/Cost Analysis) Work Plans, Sampling Plans and DQO Reports at two municipal landfills located in Toledo, Ohio. Upon approval by U.S. EPA and Ohio EPA project involvement has included: design of leachate and groundwater wells, oversight of field activities and report preparation.

Ryder Truck Rental Inc., Miami, Florida (1989-1992): Served as Project Manager, Program Director, and National Account Manager for a nationwide truck transportation firm's underground storage tank (UST) program. During a four-year period of time more than 400 projects were completed. Project involvement has included: Phase I property acquisitions, UST removals, subsurface contamination assessment, free product recovery, soil and groundwater remediation and Petroleum Reimbursement Fund applications. As part of this program, more than 15 WCC field offices were used to assist in project work throughout the United States and Canada.

Filter Manufacturing Association, Washington, D.C. (1985): Worked with a coalition of 15 oil filter manufacturing firms to complete an assessment of used waste oil filters. During this study, an innovative method of sampling filters was developed to collect representative samples from used filters. Results from the study were submitted to U.S. EPA in Fall 1991 to exempt used oil filters as a hazardous waste.

Montville Sanitary Landfill, Medina, Ohio (1985-1987): Served as Project Manager and assisted in the preparation of a Remedial Investigation/Feasibility Study for an industrial waste/sanitary landfill located in northeastern Ohio. Worked extensively with the EPA HELP model to study leachate generation.

Professional Societies/Affiliates

Association of Engineering Geologists
Cleveland Engineering Society
Association of Groundwater Scientists and Engineers

Publications

Northern Ohio Geological Society, "Remote Sensing of Earth Resources". An inventory of peatlands in northeastern Ohio using landsat imagery (with R.G. Craig, M. Dougherty). Vol. 8, pp. 215-225. University of Tennessee Space Institute. 1979.

"Compass". Shoreline erosion at Painesville Township Park, Lake County, Ohio. Vol. 57, No. 3, pp. 89-100. 1980.

"A Geotechnical and Hydrogeological Investigation of Wastewater Treatment Sludges and River Sand to be used as Sanitary Landfill Caps". Doctoral dissertation. Kent State University. Kent, Ohio. 1985.

"NWWA Proceedings, Practical Applications of Groundwater Models". Predicted landfill cap behavior of soils amended with municipal waste sludges using the HELP model (with M.R. McComas and A. Shakoor). Columbus, Ohio. Summer 1985.

"Bulletin of the Association of Engineering Geologists". Lime Sludge Stabilization of Sand for Capping Sanitary Landfills (with Abdul Shkoor) Vol. 26, No 2, pp 227-240. 1989.

"Dredging and Dewatering of Hazardous Impoundment Sediment Using the DryDREdge™ and Geotubes". (with ML. Duke, J. Fowler, and AC Askew) Western Dredging Association, Journal of Dredging Engineering, Volume 2, No. 1. March 2000.

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Presentations

Geotechnical and Hydrogeological Evaluation of Alternative Soil Materials for Capping Landfills. Presented at the Association of Engineering Geologists, Fall Meeting. Winston Salem, North Carolina. 1985.

Application of the Data Quality Objective Process in Developing Risk Based Corrective Action Remedial Strategies of Two Ohio Refineries. API Spring Refinery Symposium, San Francisco, California, 1998.

Depositions and Expert Testimony

Kay Bettis et.al. vs. Ruetgers Nease Chemical Co., Inc., et al. United States District Court Northern District of Ohio Case No. 4:90CV-0502, 1992.

Highpoint Truck Terminal, Inc. et.al. vs. Phoenix Construction Company et.al., Case No. CV93-03-0868, 1994.

BP Oil vs. Glod No. 1 Oil Well, 1995.

Ferris Inc. vs. Ashland Oil Inc. et. al., Case No. 5:94CV1303.

Contact Information

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direct line: 216.622.2432; main line: 216.622.2400; fax: 216.622.2464;
and cell phone: 216.543.2168

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James Anderson

Project Manager

Overview

Mr. Anderson has been involved in environmental, geotechnical and geological projects since 1983. His experience includes all aspects of professional practice from proposal preparation, field coordination, report preparation and overall project management. Areas of expertise include Environmental subsurface investigations; remediation system design; UST and AST system design, installation oversight; UST closures and reporting; Health & safety; and ASTM Phase I Site Assessments. Mr. Anderson's background also includes five years of petroleum and geothermal well-site geological consulting services.

Areas of Expertise

Geological
Environmental

Years of Experience

With URS: 6 Years
With Other Firms: 15 Years

Education

BS/Geology/1983/Ohio University

Registration/Certification

Year/Certified Underground Storage Tank Installer/State of Ohio/#

Project Specific Experience

Cleveland Office Health and Safety Representative, URS, Years: Responsible for implementing URS corporate health and safety policies and procedures in the Cleveland Office. Review and prepare health and safety plans. Maintain database and monitor training requirements.

Project Manager, START USEPA Emergency Responder, Location: Project Manager for USEPA 24 hour on-call Emergency Response Program. URS is a sub-contractor to the Prime contractor in Region 5. Responsible for managing, training and responding to various emergency response projects in support of Prime Contractor.

Project Manager, Petroleum Pipeline Spill, Franklin Township, Ohio, BP Pipelines: Project Manager for BP pipeline spill remediation project. Manage operation and maintenance of five remediation systems and perform all necessary reporting requirements. Interact with affected property owners, pipeline operators, BP and regulatory agencies.

Operations Manager, Remediation Implementation, Sandusky, Ohio, NASA Plumbrook Facility: Operations Manager for implementation of remedial activities at two NASA Plumbrook RCRA units. Activities included coordination of all field activities for installation of two treatment systems, profiling of soils for disposal, preparation of bid specifications, contractor oversight, and O & M preparation. remedial technologies included soil vapor extraction, vacuum-enhanced groundwater recovery and carbon absorption.

Role, Feasibility Study, Riverview, Michigan, BASF Corporation: Prepared Feasibility (FS) Study for a landfill in Michigan. The FS was prepared to determine a cost-effective way to prevent mercury contaminants from impacting waters of the State. The FS included: No action, pump & treat, capping, slurry walls, sheet pile walls, dig and haul and phytoremediation using hybrid poplar trees.

Field Manager, BP Products North America Inc., Multiple UST Sites, Ohio, Pennsylvania and Indiana: Field manager for projects at more than 50 petroleum retail sites. Projects have involved UST closures; multiple phases of environmental site assessments; slug testing; pilot testing including groundwater pumping, vacuum-enhanced groundwater extraction, dual-phase extraction and soil vapor extraction; preparation of remedial action plans; completion of remedial designs; remediation system permitting, installation, operation, maintenance, monitoring, and compliance reporting; LNAPL recovery; quarterly groundwater monitoring; and monitoring of natural attenuation of petroleum hydrocarbon compounds. Remedial technologies have included soil removal, application of oxygen release compound, soil vapor extraction, vacuum-enhanced and conventional groundwater recovery, air stripping and carbon absorption.

Project Manager and UST Group Leader, UST, Cleveland, Ohio: Manage UST sites throughout Ohio. Projects in various stages from UST closures through Tier 1 and Tier 2 Assessments. Clients include: City of Garfield Heights, The City of Maple Heights, Longaberger, Yellow Freight, Sears, International Paper and others.

Field Operations Manager, RCRA Closure Assessment, Sandusky, Ohio, NASA Plumbrook Facility: Field Operations Manager for a RCRA Closure Assessment at the NASA Plumbrook research Facility, which was a former military munitions facility in Sandusky, Ohio. Responsibilities included coordination of all field activities for a four-month soil and groundwater assessment.

Project Manager, Gas Well Oversight, Location, Marconi Medical: Performed oversight and regulatory review for the installation of a gas production well. Duties included regulatory review of permitting process, review of drilling and production activities for compliance with state regulation and environmental issues and recommendations for future O&M activities.

Project Manager, Multiple UST Sites, Utah, Utah Department of Transportation: Project Manager for Utah Department of Transportation UST/AST program, which included: design and oversight of UST and AST system installations; UST and AST closures; subsurface investigations and reporting; remediation design and implementation, for sites throughout Utah.

Field Manager, Dugway Proving Grounds, Dugway, Utah: Field Manager for UST program at a US military facility, which included the inspection of 25 UST systems for compliance with state and federal regulations, prepared recommendations and specifications for upgrades and closures.

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Project Manager, ASTM Site Assessments, Ohio: Project Manager for numerous ASTM Phase One Site Assessments and Phase II Site Assessments throughout Ohio.

Senior Partner, Well Site Consulting Service, Utah: Senior Partner for oil field well site consulting services on numerous shallow to deep exploration and production wells throughout Uintah Basin, Utah. Staffed and maintained several mobile lab units. Responsibilities included the management of a small business.

Independent Well Site Geologist, Rocky Mountain Region: Independent Well Site Geologist for oil field production and exploration wells throughout the Rocky Mountain Region. Responsibilities included the collection of drill cuttings, monitoring of drilling fluids and formation gases, and the documentation of all drilling activities.

Geothermal Well Site Geologist, China Lake, California: Well Site Geologist for large geothermal drilling and production project in the China Lake Region, California. Responsibilities included the monitoring of formation temperatures and pressures, the collection of drill cuttings, the documentation of drilling activities, and locating the placement of casing points.

Professional Societies/Affiliates
Geological Society of America

Awards
[Click **here** and type Year/Award Name/Awarded by]

Languages
[Click **here** and type Language(s)]

Specialized Training
[Click **here** and type Year/Training Course]

Security Clearance
[Click **here** and type Security Clearance Level]

Publications
[Click **here** and type "Article Name", Publication, Vol #, Month, Year]

Chronology
[Click **here** and type Dates 00/00-00/00: Company Name, Location]

Contact Information
[Click **here** and type Address, Phone Fax, E-mail Address]

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Peggy J. Schuler

Senior Environmental Scientist

Overview

Ms. Schuler has over 20 years of experience in analytical chemistry, environmental testing, and environmental regulations, specializing in various types of sample analyses, Quality Assurance/Quality Control (QA/QC), data validation, and data management. Her experience includes over eight years of direct bench experience in the analysis of waters, soils, and wastes for inorganic parameters, as well as seven years of experience in the assessment of environmental data. She has a comprehensive knowledge of laboratory quality assurance/quality control and analytical methodologies, especially SW-846, MCAWW, and USEPA CLP methods. She is proficient in data validation, technical writing, and the use of MS Access for data management. In addition, her duties have included supervision and training of laboratory analysts, coordination of laboratory and field activities, project management, and risk assessment.

Areas of Expertise

Analytical Chemistry
Environmental Testing
QA/QC

Years of Experience

With URS: 6 Years
With Other Firms: 15 Years

Education

BS/Zoology/1981/Michigan State University
Post-Baccalaureate Courses in Computer Science and Business/ University of Akron (1982-85)

Registration/Certification

1987/Wastewater Laboratory Analyst, Class III/Ohio/3-87-22

Project Specific Experience

Risk Assessor, Phase II Property Assessment, Tiffin University, Tiffin, OH: Performed a human health risk assessment in accordance with Ohio's VAP. Responsibilities included identification of potential exposure pathways and receptors and evaluation of risk, assuming future residential and commercial land use scenarios as defined by the VAP.

Project Chemist, Coal Ash Reuse Consulting, AES Hawaii, Inc., Honolulu, HI: Provided consulting services concerning reuse options and permitting requirements for fluidized bed combustion ash generated by a coal-fired electric energy facility on the island of Oahu. Responsibilities included investigation of local zoning and solid waste management regulations, review of existing ash and groundwater data, development of a work plan for an ash amendment study, and coordination of laboratory analyses.

Project Chemist, Groundwater Monitoring Plan Implementation, C&D Disposal Technologies, LLC, Wintersville, OH: Developed sampling and analysis schemes for two facilities to comply with Ohio groundwater monitoring requirements for municipal solid waste and construction and demolition debris landfills. Responsible for sampling coordination with the laboratory, data review and management, and reporting to OEPA per the approved monitoring plans.

Project Chemist, Phase II Property Assessment, Flats East Bank, Cleveland, OH: Responsible for data management and sampling coordination with the laboratory for an extensive data collection effort in support of a brownfield redevelopment project. Evaluated the analytical data for quality and usability, then performed statistical calculations for a human health risk assessment in accordance with Ohio's VAP.

Document Control Coordinator and Trainer, ISO 14001 Implementation, International Steel Group Inc., Cleveland, OH: Coordinated production of the ISG corporate EMS Implementation Guide in support of the company's ISO 14001 certification process. Assisted in development of training materials for presenting ISO 14001 Awareness and EMS Implementation Training. Provided assistance to ISG Cleveland Works personnel in the development and implementation of their Environmental Management System.

Project Chemist, Coal Ash Leachate Containment Study, Duke Energy Generation Services, LLC, Raleigh, North Carolina: Developed a bench-scale amendment and analysis program to evaluate treatment options for reducing leachable contaminants in coal ash. Coordinated sample preparation and analyses at multiple laboratories. Responsible for data assessment, management, interpretation, and graphic presentation.

Project Manager, Beneficial Ash Reuse Project, Confidential Client: Planned and executed a strategy to obtain regulatory approval for the use of coal ash as a product additive. Responsibilities included investigating coal ash disposal and reuse policies, developing a sampling and analysis plan, coordinating field and laboratory activities, assessing analytical data quality, evaluating compliance with regulatory requirements, and preparing a Permit to Install (PTI) Application package for submission to Ohio EPA.

Data Validator, BP Exploration and Oil, Inc., Lima Refinery, Lima, Ohio and BP Exploration and Oil, Inc., Toledo Refinery, Toledo, Ohio: Perform ongoing data quality assessments and some data management in support of RCRA Facility Investigations and Closure activities. The projects involve(d) validation of analytical data for both soils and waters for volatiles, semivolatiles, pesticides, PCBs, herbicides, metals, and selected inorganic parameters, for the purposes of site characterization and decision-making.

Project Chemist, Proposed Golf Course Ash Reuse Project, Dominion Energy, Chesapeake, Virginia: Performed data assessment and management for fly ash stabilization studies for the purpose of evaluating land application options in the development of a proposed golf course. The project included extensive groundwater modeling to determine the maximum contaminant concentrations (and the associated risk) in the groundwater at the nearest downgradient receptors over a 150-year span.

Project Chemist, Chicago Coke Plant Decommissioning, LTV Steel Company, Chicago, IL: Developed a sampling and analysis strategy for disposal of process chemicals and residues throughout the Chicago Coke Plant. Designed and maintained an Access database in support of a Geographical Information System for presenting the data generated

during the decommissioning process. Assisted in project coordination and information management.

Project Chemist, Gentile Air Force Station, Kettering, Ohio; Rickenbacker Air National Guard Base, Columbus, Ohio; and Grissom Air Force Base, Indiana: Performed various data quality/management duties in support of Remedial Investigations and/or Long-Term Monitoring at three closed Air Force facilities under the Installation Restoration Program. Responsibilities include: Preparation and/or review of project documents, including Work Plans and Quality Assurance Project Plans; procurement and coordination of laboratory services; assessment and documentation of data quality and usability; and preparation of electronic deliverables in ERPIMS database format to report geological, physical, and chemical data associated with sampling activities at the site.

Project Chemist, Hexavalent and TCLP Chromium Study, Confidential Client: Developed a sampling and analysis strategy to supplement historical data for two wastestreams at a manufacturing facility, for the purpose of characterizing the wastestreams in regard to the various chromium fractions—total, hexavalent, and TCLP. This included a review of over ten years of existing data for quality and usability, and identification of data gaps. The sampling, analyses, and statistical evaluation were performed in accordance with "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods [SW-846]," (USEPA, 1986). Additional responsibilities included coordination of the sampling activities and laboratory services, and preparation of the project report.

Data Validator, West Virginia Ordnance Works Operable Unit-13 (OU13), Pantasote Site, Point Pleasant, West Virginia: Performed document review and data validation in support of the Engineering Evaluation/Cost Analysis (EE/CA) at the OU13 Superfund site in Mason County, WV. The project involved assessment of analytical data for both soils and waters for volatiles, semivolatiles, pesticides, PCBs, dioxins, metals, and selected inorganic parameters, for the purpose of selecting alternatives for reducing or preventing the release of contaminants from OU13.

Project Chemist, BASF Corporation, Riverview Site, Wayne County, Michigan: Coordinated laboratory services and acted as a liaison between BASF and the laboratory for assessment activities initiated by the Michigan Department of Environmental Quality (MDEQ) at the Riverview landfill site. Also assessed the quality of the laboratory data and its usability to support decision-making concerning cleanup options at the site.

Technical Reviewer, Naval Facilities Engineering Service Center (NFESC), Environmental Services Department: Pre-qualified environmental laboratories for Navy contracts by assessing laboratory data and standard operating procedures according to NFESC guidelines.

Also performed technical reviews of Quality Assurance Project Plans (QAPPs) for environmental investigations at several Naval installations.

Data Validator, New Jersey Department of Environmental Protection (NJDEP), Hudson County, New Jersey: Provided data validation services to NJDEP for the remedial investigations of the Hudson County Chromate Chemical Production Waste Sites. Used professional judgement and NJDEP guidelines to assess the usability of analytical data for delineating the extent of contamination from historical chromite ore processing operations, and for identifying and evaluating cleanup options.

Database Analyst and Trainer, LIMS Implementation, Ross Analytical Services, Strongsville, Ohio: Assisted in the evaluation, selection, and customization of the Horizon LIMS by Chemware, including analyzing work flows and user requirements in each department, defining and mapping the individual procedures comprising each analysis, and collecting the information required to populate the underlying control tables. The project also included developing and conducting user training programs for laboratory, sales, and management personnel.

Laboratory Quality Assurance: Authored/reviewed/updated the laboratory Quality Assurance Manual and Standard Operating Procedures for a full-service environmental laboratory. Assisted in internal audits, analyst training, and certification activities. Responsible for performing and/or supervising the final review of all analytical data generated by five laboratory departments, and generating client reports and data deliverables. Duties required a thorough knowledge of laboratory capabilities, analytical methodologies, and federal and state regulations under BUSTR, OEPA, RCRA, CLP, and others.

Analytical Experience: Includes metals analysis by atomic absorption and inductively coupled plasma, as well as many conventional analytical procedures including colorimetric, gravimetric, titrimetric, chromatographic, and other instrumental methods.

Chronology

05/98 – 11/99	Quality Specialists and Environmental Analysts, Inc., Sheffield Lake, Ohio
02/91 – 03/98	Ross Analytical Services, Inc., Strongsville, Ohio
09/87 – 01/91	Envirotech Operating Services, Inc., Strongsville, Ohio
09-85 – 09/87	City of Akron, Water Pollution Control Station, Akron, Ohio

Contact Information

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peggy_schuler@urscorp.com

URS

David I. Blaushild

Laboratory Program Manager

Areas of Expertise

- Quality Assurance Management
- Data Management
- Environmental Laboratory and Field Sampling Auditor
- Analytical Procurement
- Data Validation and Usability
- General Environmental Chemistry

Years of Experience

With URS: 4 Years
With Other Firms: 26 Years

Education

State University of New York, College of Environmental Science & Forestry (at Syracuse University): BS, Chemistry, 1975
Syracuse University: graduate-level business courses
St. Johns University: Graduate-level chemistry courses

Registration/Certification

OSHA 40-hour Hazwoper Training and 8-hour Annual Refresher

OSHA 8-hour Hazwoper Supervisory Training

Overview

Mr. Blaushild has over 29 years experience in the quality assurance management and data management field. He is also experienced in polymers and plastics, pharmaceuticals and research biochemical industries in the areas of organic synthesis, quality control and pilot plant production, industrial hygiene and general environmental consulting.

Mr. Blaushild is proficient with a wide variety of laboratory instrumentation and chemical techniques including spectroscopy, chromatography, physical testing instrumentation; wet chemical analyses; and a number of laboratory computer systems. He has extensive experience with hazardous waste, drinking water, wastewater, and air analytical procedures; and various sampling techniques.

Mr. Blaushild has extensive knowledge of regulations including CERCLA, RCRA, CWA and OSHA.

As the lead chemist and client contact for a large chemical distribution company he has prepared, implemented and manages the environmental laboratory management program.

He possesses a thorough knowledge of quality assurance/quality control (QA/QC) project management.

Mr. Blaushild is currently manager of the URS Diamond Analytical Quality and Data Management functions. He is responsible for laboratory coordination, data review and management, and other environmental chemistry issues for all Ashland environmental remediation projects.

Project Specific Experience

- As Senior Chemist for the Environmental Strategies Corporation (ESC), acted in capacity of chemical expert for environmental consulting firm including interpretation of material safety data sheets; educating clients and staff on environmental chemistry and data management. Developed and presented various in house training courses on chemistry for non-chemists.
- Prepared work plans and reports for environmental investigations including field-sampling plans, health and safety plans, quality assurance project plans, data validation, quality assurance summary reports, and sampling event summary reports.

- Performed numerous field sampling and laboratory on-site audits and presented recommendations for corrective action to management.
- Developed working knowledge of environmental regulations including RCRA, CERCLA, CWA and various state voluntary clean up programs.
- Responsible for negotiating with laboratories and state and federal environmental agencies on behalf of clients.
- Prepared the clients' laboratory contracting and corrective action procedures manual to standardize the sample collection and analysis procedures saving the company over $250,000. Developed and maintained ESC's partner laboratory program.
- As a Program Manager with the Center for Hazardous Materials Research, organized and managed large-scale production of biofiltration media; responsibilities included qualifying raw material suppliers, coordinating and managing production, and testing product.
- Performed pilot scale testing of solid waste composting systems and developed working knowledge of solid waste regulations in various states.
- Provided technical support and training to sister company and organized and assisted in setting up a bioremediation testing facility. Responsible for testing bioremediation related products on a large US EPA field study. Supervised and trained three technicians.
- Managed Quality Assurance program for environmental fate pesticide testing laboratory under USEPA GLP regulations.
- Supervised and trained three technicians.
- As a Project Manger for Pace, Inc. managed large environmental laboratory projects for private clients and public agencies. Provided technical support to industrial clients. Supervised a data management and data reporting group.
- Managed quality assurance program for environmental testing laboratory. Negotiated with government agencies on behalf of clients.
- As a chemist with CS Environmental analyzed air, soil, and water samples for environmental contaminants using GC/MS, GC and HPLC. Supervised the work of two technicians. Evaluated, purchased and installed laboratory instrumentation and equipment.
- As a Research Scientist with Bristol Myers Squibb, performed bench and pilot scale production of pharmaceutical products including process improvement. Evaluated fermentation products

URS

Chronology

2002 to Present
URS Corporation,
Senior Project Chemist,

1993-2002
Environmental Strategies
Corporation, Senior Chemist,

1990–1992
Center for Hazardous Materials
Research, Quality Assurance
Officer/Project Manager,

1988–1990
Pace, Inc., Project Manager/Quality
Assurance Manager,

1986–1988
CS Environmental, Inc., Chemist,

1983-1986
Bristol Myers-Squibb, Inc., Research
Scientist,

1979–1983
DuPont Company, New England
Nuclear Division, Chemist,

1976–1979
Occidental Chemical Corporation,
QC Chemist/Technical Services,

for production of pharmaceutical intermediates including antibiotics and antitumor compounds.

- Improved productivity by increasing product yield and stimulating metabolism rate of microbes. Studied secondary metabolism of fungi and bacteria. Analyzed pharmaceutical intermediates and raw materials utilizing HPLC, GC, FTIR and UV-Vis.
- Set up a laboratory data information management system.
- As a Research Scientist with DuPont/NEN Products developed new biochemical intermediates and new products relating to biotechnology, including immobilized enzymes on solid supports and radioactive isotopes used in biological research. Synthesized novel organic materials. Analyzed products Utilizing HPLC, GC, UV-Vis, IR and autoanalyzers.
- Assisted in waste reduction program and radiation waste program.
- As a Technical Services and QC Chemist at the Occidental Chemical Corporation conducted industrial hygiene monitoring of plant workers to evaluate potential exposures to hazardous chemicals. Evaluated continuous monitoring devises for plant emissions.
- Responsible for quality control and process improvement for polymers and plastic compounding facility including evaluation of raw materials, finished products, and competitor product evaluation.
- Provided technical support to end users of company's products, including troubleshooting and site visits.

Affiliations

- American Chemical Society
- Air & Waste Management Association

URS

Douglas R. Gray

Principal Project Manager / Innovative Remedial Technologies Group Manager

Areas of Expertise

Project Management

Remedial Design/Construct

RCRA/CERCLA Site Assessments

PADEP Act 2

Innovative Remedial Alternatives Design, Evaluation, and Implementation

Data Visualization

Groundwater Modeling

Years of Experience

With URS: 14 Years

With Other Firms: 9 Years
(Dow Chemical Company)

Education

The Ohio State University: BS Chemical Engineering - 1984

Technical Specialty - Reservoir Eng.

The Ohio State University/NGWA: Credential in GW Science-1992

Registration / Certification

Association of Groundwater Scientists and Engineers (AGWSE)

Specialized Training

PA Department of Environmental Protection – 2003 Land Recycling Program (Act 2) Client Workshop

OSHA 40-Hour Health and Safety Training for Hazardous Waste Site Workers and Operations

OSHA 8-Hour Hazardous Waste Site Supervisory Training

OSHA 30-Hour Construction Safety Training

URS Project Management Certification Training

Multiple GW Assessment and Remediation Training Classes
(List available on request)

Overview

Twenty-two (22) years of project management and engineering experience focusing on the development, design, costing and optimization of state-of-the-art, innovative remedial alternatives, site assessments, remedial alternative evaluations, remedial investigations, and feasibility studies. Project experience also includes the development of computer groundwater databases for categorizing and assessing site geologic, hydrogeologic, and chemical conditions, as well as the development of computer groundwater models for both flow and solute transport.

As a Project Manager and Innovative Remedial Technologies Group Manager, responsibilities cover a wide range of environmental engineering, personnel management, budget planning, and marketing duties. Some of these responsibilities include hydrogeologic assessment and field supervision, development of work plans, sampling and analyses plans, remedial investigations and feasibility studies, preparation of proposals and cost estimates, design/costing/optimization of state-of-the-art innovative remedial technologies; design/implementation of natural attenuation and other innovative in-situ remedial alternatives, participation in local/state/federal agency negotiations, business development and marketing of commercial and government clients related to a variety remediation approaches, as well as the development of computer groundwater models for both flow and solute transport related to hydrogeologic and remedial investigations.

Also knowledgeable and experienced in the internal training of personnel related to environmental engineering and issues, RCRA and CERCLA regulatory environments, as well as participating as both a presenter and Session Chair (Battelle Conference) for a variety of technical symposiums.

Selected Project Specific Experience

Innovative Remedial Technologies Group Manager
2003 - Present
Serving as the Group Manager for the Innovative Remedial Technologies Group, located in Pittsburgh, PA, for the past three (3) years. This group specializes in the evaluation and implementation of innovative remedial technologies including ISCO, enhanced bioremediation, enhanced free product recovery, zero-valent iron, emulsified vegetable oil, and other technologies geared towards providing a more cost effective remedial solution for the client. The technologies are often more beneficial and cost effective for the client compared to the more traditional/long-term remedial methods that have been typically employed in the past.

PA Department of Environmental Protection, Site Remediation and Groundwater Treatment System O&M at a former Service Station Release Site, PA
2004 - Present
Project Manager for the oversight and O&M functions associated with a large groundwater treatment plant that was installed to provide for the pump and treat remediation of a BTEX / MTBE plume resulting from the release of a

large quantity of fuel from a leaking UST. This project involves interacting with the on-site O&M personnel, state regulatory agencies, and other local individuals in order to maintain the treatment plant operations and to implement other activities at the Site as directed by the state agency. The remediation is being conducted to clean up the site to PADEP Act 2 standards.

PA Department of Environmental Protection, Site Characterization Investigation and Remedial Action Plan at a former Service Station, PA
2003 - Present
Project Manager / Technical Lead for a remedial investigation and remedial action plan under PADEP Act 2 Program. The subsurface is contaminated with BTEX and MTBE to a depth of approximately 110 ft-bgs. Plume characterization, fate and transport modeling and remedial alternative evaluations completed under Act 2. Remedial action consists of oxygen addition to provide for enhanced in-situ biodegradation and continued groundwater monitoring to support the natural attenuation of constituents that were formerly present off-site on adjacent properties. Additionally, the project requires the O&M of operating GAC and RO units located at adjacent residential locations and the interaction with local residents to discuss ongoing remediation activities.

Confidential Chemical Client Feasibility Evaluation / Implementation of Emulsified Vegetable Oil Remediation, NJ
2004 - Present
Project Manager for the evaluation, feasibility assessment, and implementation of an enhanced remedial approach at an active chemical facility using emulsified vegetable oil to provide for enhanced reductive dechlorination of site contaminants. Contaminants had been reduced to just above regulatory concern levels using traditional SVE and dual-phase extraction remedial technologies. Prior remedial efforts reached asymptotic levels and client desires to receive a No Further Action (NFA) with regard to this remedial system. Additional site data review and sampling activities indicated that contaminants were undergoing in-situ biodegradation. Based on the available data emulsified vegetable oil was introduced into the subsurface to provide for enhanced biodegradation through the reductive dechlorination of site contaminants. Existing infrastructure from an existing remediation system was utilized to introduce the emulsified vegetable oil in a cost effective manner for the client. The project field work was completed in January 2006 and performance monitoring is currently underway with groundwater data indicating highly reducing conditions, and the initial decrease in groundwater contaminant levels has been observed.

Confidential Chemical Client Feasibility Evaluation / Implementation of Emulsified Vegetable Oil Remediation, IN
2004 - Present
Technical Lead / Task Manager for the evaluation / feasibility assessment and implementation of an enhanced remedial approach in a 30-acre parcel of a former laboratory/testing facility using emulsified vegetable oil to provide for enhanced reductive dechlorination of site contaminants. Contaminants are present in a sand & gravel zone located within a glacial till. Prior remedial efforts focused on pump and treat operations. A pending site divestiture prompted client to desire an alternative remedial approach. Additional site characterization activities and sampling indicated that contaminants were undergoing in-situ biodegradation. A microcosm study further indicated that enhanced biodegradation could be realized

through the addition of emulsified vegetable oil to provide a carbon source, nutrients, and promote highly reducing groundwater conditions. The project field work was completed in November 2005 and performance monitoring is currently underway with the existing data showing promising results.

Confidential Chemical Client PADEP Act 2 Implementation / Feasibility Evaluation Chemical Oxidation Pilot Study at Chemical Facility, PA
2003 - Present
Technical lead / task manager for the evaluation and feasibility assessment of entering the Facility into the Act 2 Program pending a divestiture of the site. Existing data was evaluated, data gaps identified, and recommendations on how to proceed along with a best case exit strategy from the Act 2 Program were developed and presented to the client in a Technical Memorandum.

PA Department of Environmental Protection, Site Characterization Investigation and Remedial Action Plan at a former Service Station, PA
2002 - Present
Serving as peer reviewer for a remedial action plan under PADEP Act 2 Program. The subsurface is contaminated with BTEX and MTBE to a depth of approximately 18 ft-bgs. Plume characterization, fate and transport modeling and remedial alternative evaluations completed under Act 2. Remedial action consists of in-situ chemical oxidation and in-situ oxygen addition to address both the BTEX and MTBE plumes. Additionally served as peer reviewer for the design, implementation, and data interpretation of an in-situ chemical oxidation remediation at a service station. Pilot study was successful and a full-scale remediation was implemented to clean up the site to PADEP Act 2 standards.

PA Department of Environmental Protection, Site Characterization Investigation and Remedial Action Plan Study, PA
2002 - Present
Peer Review / Technical Lead for the design, implementation, and data interpretation of a site characterization and remedial action plan development under PADEP Act 2 Program. The subsurface is contaminated with BTEX and MTBE to a depth of approximately 80 ft-bgs. Plume characterization, fate and transport modeling, and remedial alternative evaluations have been completed under Act 2. Ongoing SVE operations, additional groundwater recovery wells, and other innovative remedial technologies and natural attenuation evaluations are presently being completed.

Confidential Client, In-situ Chemical Oxidation Pilot Study at Former Refinery Facility, OH
2002 - 2004
Project Manager /Technical lead for the design, implementation, and data interpretation of an in-situ chemical oxidation remediation at a refinery facility. The subsurface was contaminated with petroleum hydrocarbon LNAPL and dissolved phase petroleum hydrocarbons to a depth of approximately 20 ft-bgs. The study was completed and the client decided to take another approach at the site.

Confidential Client, In-situ Chemical Oxidation at Former Chemical Facility, NJ
2001 - 2003
Project Manager / Technical lead for the design, implementation, and data interpretation of an in-situ chemical oxidation remediation at a former chemical facility. Subsurface contaminated with BTEX and chlorinated constituents to a depth of approximately 12 ft-bgs. Following the completion of field work, a six-month performance-monitoring period was completed and the successful completion of the project was reported to the NJDEP, with receipt of an NFA for the treated area.

Confidential Client, Innovative Remedial Technologies Evaluation, Preliminary Design, and Cost Estimation, Former Manufacturing Facilities, OH
2001 - 2002
Serving as technical lead as part of a multi-office team that prepared innovative remedial technology feasibility studies, preliminary remedial alternative designs, and initial cost estimates at two (2) facilities in Ohio.

Confidential Client, Innovative Remedial Technology Design/Evaluation
1999 - 2002
Project Manager related to the evaluation of an existing pump and treat groundwater recovery and treatment system focusing on the replacement of this system with innovative remedial alternatives including natural attenuation, enhanced in-situ biodegradation, permeable reactive walls, and/or source area treatment utilizing either HRC®, zero-valent iron or other in-situ chemical oxidation alternatives.

Confidential Client, RCRA Corrective Action Strategy (CAS) Program USEPA Region VI and ADEQ, AR
1998 - Present
Project Manager / Technical reviewer of historical groundwater quality data for a RCRA facility in Arkansas to evaluate natural attenuation. The primary contaminants consisted of PAH compounds. The evaluation supported the development of a presentation to demonstrate that the dissolved phase plume had reached steady state and that remediation by natural attenuation was occurring at the facility. As part of the ongoing CAS Program activities, current responsibilities include the design/evaluation of additional innovative, enhanced DNAPL recovery systems at the facility, including recovery well design, re-circulation well design, pilot testing, optimization, shakedown testing and agency interaction/planning. Presently, monthly DNAPL recovery rates average between 3000-4000 gallons per month with associated groundwater recirculation rates of approximately 90-100 gpm.

Confidential Commercial Client, LA
1995 - 1999
Project Manager / Senior Scientist responsible for project oversight, data compilation, site assessment, and subsequent development of a site-wide database and groundwater flow/transport model. Modeling was completed for the design of recovery systems utilizing 25 horizontal wells at two depths within the site. Groundwater modeling was also utilized to address risk based cleanup levels for the contaminated groundwater beneath the site.

Confidential Client, Plaquemine, LA
1995 - 1999
Project Manager / Senior Scientist responsible for project oversight, data compilation, site assessment, and subsequent development of a site-wide database and groundwater flow/transport model. Modeling work has been completed for the design of three phases of recovery systems utilizing 25 horizontal wells at two depths within the site. Groundwater modeling work at the site was also utilized to address the development of risk based cleanup levels for the contaminated groundwater beneath the site.

Confidential Client, RCRA Facility Investigation (RFI) and Corrective Measures Study (CMS), AR
1995 - 1998
Project Manager and Senior Scientist at a 200- acre wood treating site. Developed work plans, schedules, regulatory reporting, and project budget/cost tracking. Developed relational database for geologic, hydrogeologic, and chemical data, performed groundwater modeling for the CMS, and supervised the sampling of over 100 monitoring wells as part of the RFI SS and CMS.

Confidential Client, Midland, Michigan
1995 - 1996
Senior Scientist in the evaluation of data from a former landfill area. This evaluation consisted of the interpretation of a groundwater hydraulic and chemical data, which led to the development of a corrective action plan for the closed facility. Also served as Senior Scientist in the development of responses to Notices of Deficiency (NODs) with respect to the Site Operating Permit that was under review by the regulatory agencies.

Confidential Client, Sarnia, Ontario CANADA
1988 - 1994
Project manager and technical lead for the evaluation of a hazardous waste landfill and associated DNAPL contamination both within and off the site property. Designed and implemented several investigation to delineated the extent of contamination and develop remediation and closure plans for the landfill and surrounding property.

Project manager and technical lead for the design and installation of a vibrated beam slurry wall. The slurry wall was constructed using a proprietary admixture of sand, cement, asphalt, and water to provide extremely low permeability as well as resistance to organic constituents in the groundwater. In addition, an upgradient groundwater extraction system was designed consisting of nine (9) extraction wells along the 1200 foot slurry wall.

Confidential, Multiple Sites, US and Europe
1984 - 1990
Designed hydrogeologic study programs and field methods for multiple sites in North America and Europe. Evaluated the field data to yield an assessment of the subsurface conditions at the sites, delineate the extent of subsurface contamination, and evaluate potential remedial alternatives. This work included soil borings, installation of piezometers and monitoring wells, data evaluation, groundwater modeling, groundwater database development, and remedial alternative evaluations. Participated in several site environmental / groundwater audits as well as providing technical project review resources for information at two Superfund sites. Served as part of a groundwater training team in conjunction with the confidential company.

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Publications

G. Klecka, J. Davis, D. Gray, and S. Madsen, "Natural Bioremediation of Organic Contaminants in Groundwater: Cliffs-Dow Superfund Site", Groundwater, July-August, 1990, Volume 28, Number 4 Issue.

A. Nazar, D. Gray, T. Oolman, S. McDougall, J. Ferguson, and F. Coll, "Dual-Well Recovery System Design for Enhanced DNAPL Production", 1997 Petroleum Hydrocarbons Conference, National Groundwater Association; Houston, TX.

F. Coll, D. Gray, K. Paschl, M. Brourman, and M. King, "Integrated Mass Recovery and MNA for RCRA Corrective Action", 2003 Battelle Remediation Conference; Orlando, FL.

F. Coll, D. Gray, and J. Danihel, "Integrated In-situ Remedies for UST Site Remediation", 2005 Battelle Remediation Conference; Baltimore, MD.

F. Coll, D. Gray, and E. Gursky, "Incorporation of Interim Measures for Water Supply Protection: A Case Study", 2005 Battelle Remediation Conference; Baltimore, MD.

D. Gray, F. Coll, and R. Fasano, "In-situ Remedial Success at a Multiple Remedy Mixed Constituent Site", 2005 Battelle Remediation Conference; Baltimore, MD.

D. Gray, F. Coll, R. Fasano, D. Raymond, and T. King, "Persistent Chlorinated Constituent Remediation with Emulsified Oil Utilizing Existing Infrastructure", 2006 Battelle Remediation Conference; Monterey, CA.

D. Gray, F. Coll, K. Paschl, and M. King, "Mass Discharge Flux Estimation to Support Corrective Action at a Creosote Site", 2006 Battelle Remediation Conference; Monterey, CA.

D. Gray, F. Coll, J. Powers, K. Gaskill, D. Raymond, and T. King, "Enhanced In-situ Bioremediation of Chlorinated VOCs as a Cost-Effective Remedial Alternative", 2006 Battelle Remediation Conference; Monterey, CA.

Employment Chronology

URS Corporation - Principal Project Manager, URS Pittsburgh, PA Office April 1992-Present

The Dow Chemical Company - Senior Environmental Engineer, Strongsville, OH 1984 – April 1992

Address / Contact Information

Douglas R. Gray

Work Contact Information:

URS Corporation
Foster Plaza 4
501 Holiday Drive, Suite 300
Pittsburg, PA 15220
Phone: (412) 503-4671

URS

Frederic R. Coll

Principal Geologist

Areas of Expertise

Project Management

Hydrogeologic Evaluation/Investigation

Regulatory Interaction

Innovative Remedial Design and Implementation

Enhanced DNAPL Recovery

Geochemical Characterizations to Support MNA

Years of Experience

With URS: 10 Years

With Other Firms: 1 Year

Education

Kent State University: MS, Engineering Geology-1994

Slippery Rock University: BS, Geology-1992

Registration / Certification

Registered Professional Geologist in Pennsylvania, 1999

Specialized Training

40 Hour OSHA Hazardous Waste Operations

8 Hour OSHA Hazardous Waste Site Supervisor Training

OSHA 40-Hour Health and Safety Training for Hazardous Waste Site Workers

OSHA 8 Hour Supervisory Training

Overview

Mr. Coll has over 11 years experience with the implementation and management of many aspects of environmental engineering including site assessments, site characterizations, hydrogeological investigations, geochemical investigations, geophysical investigations, geotechnical modeling, data interpretation and reporting, and site remediation. Mr. Coll also has experience with innovative remedial designs for free product and groundwater extraction and treatment systems, and monitored natural attenuation evaluations. Mr. Coll has also completed various geotechnical studies including slope stability, settlement analysis, and storm water management. He has managed and directed numerous field investigative and innovative remediation programs.

Selected Project Specific Experience

Lead Hydrogeologist
Implementation of an enhanced in-situ bioremediation project at a former chlorinated solvent site in New Jersey. Activities included oversight, data evaluation and reporting, and Agency negotiation.

Technical Lead and Project Hydrogeologist
DNAPL recovery program at a RCRA Facility in Ohio. Major activities included oversight of soil borings completed to obtain data on DNAPL extent. This data, along with data obtained through an extensive review of previous investigations at the Facility, was used to locate the recovery well. A thorough recovery well design as completed in an attempt to optimize the recovery of DNAPL. Following well installation, three recovery scenarios were implemented utilizing different pumping schemes to determine the best recovery setup. In addition to DNAPL recovery testing, intensive aquifer testing was completed to determine hydro-geological parameters. This information is currently being used to design a long-term recovery system.

Project Manager and Technical Lead
Authored and sealed several Site Characterization Reports (SCRs) and Remedial Action Plans (RAPs) under the PA Act 2 Land Recycling Program. Also served as Project Manager and Technical Lead for numerous projects, including the implementation of three (3) RAPs in PA.

Project Manager
Managing several LUST sites under a PADEP contract. Activities included site characterizations, innovative remedial technology screening, pilot testing, and innovative remedial implementation including in-situ chemical oxidation and enhanced bioremediation.

Project Manager
Served as Project Hydrogeologist for a DNAPL site in northeastern Ohio. Activities included historical data evaluations and conceptual site model development in order to determine the efficacy of past source removals to support a due diligence effort at an active chemical manufacturing facility. Activities included presentations to the regulating Agency and neighboring facilities that ultimately resulted in significant cost savings to the client as it was determined that historical remedial efforts were inadequate, resulting in the re-impacts of DNAPL to a flood plain.

Project Manager and Technical Lead
Site characterization and remedy implementation at a former LUST site near Pittsburgh, PA that includes BTEX and MTBE plumes threatening a public water supply. Remedy includes operation of a refurbished SVE system and a strategic groundwater monitoring program including MNA evaluation.

Project Manager and Technical Lead
Innovative remedy implementation at a former LUST site near Wilkes-Barre, PA. Remedy includes the combined implementation of two innovative remedial technologies including in-situ chemical oxidation and oxygen infusion / enhanced biodegradation to remediate soil and groundwater impacts in a sensitive community.

Lead Hydrogeologist and Site Manager
For a RCRA site in Arkansas. Responsible for the design, installation, hydraulic testing, and development of an operation and maintenance plan for a DNAPL recovery system utilizing groundwater re-circulation. Designed and installed four (4) enhanced DNAPL recovery wells which realize a total of approximately 75 gallons per day (gpd) of creosote DNAPL recovery. Currently serve as O&M oversight and troubleshooting for local O&M contractor. Also serve as technical lead for the groundwater monitoring program at the site. Activities include data evaluation with natural attenuation documentation, regulatory interaction, and reporting. The site is one of US EPA's Corrective Action Showcase Pilot sites.

Peer Review
Performed Peer Review of Phase II Investigation Reports, Remediation Work Plans, and Remediation Completion Reports for the Indiana Department of Environmental Management for applicants pursuing clean closure under the Indiana Voluntary Remediation program.

Technical Lead
For a focused hydrogeological evaluation of a confidential chemical manufacturing facility in north Georgia impacted by chlorinated compounds. Responsibilities included development and implementation of a direct push sampling and a MNA evaluation.

Served as Site Manager and Technical Lead
Resistivity survey at an active Air Force Base in Wyoming. The resistivity survey was conducted following an electromagnetic survey to determine the depth of suspected fill areas and the groundwater depth, to support remedial activities.

Served as Task Leader
Environmental investigation in northwestern PA for a confidential client. Performed historical document review, field investigation activities, and reporting for a law firm representing a Pennsylvania municipality in a cost recovery lawsuit. Project duration was seven months with a final deliverable of an Expert Witness Testimony Report.

Project Engineer
Performed various geotechnical stability and surface water conveyance calculations and design for a chemical manufacturing waste landfill in western Ontario, Canada. Activities included cap design, surface conveyance, and slope stability. Additional activities included stability evaluation for excavation along an existing sheet pile wall and subsequent determination of the minimum acceptable penetration of piles required for cantilevered stability.

Project Hydrogeologist
DNAPL recovery program at a RCRA Facility in Ohio. Major activities included installation of soil borings and monitoring wells to support the design of a DNAPL recovery well in bedrock.

Project Hydrogeologist
Assisted in the preparation of a Mixing Zone Application for an active chemical manufacturing Facility in South Carolina. Activities included review of 10 years of previous analytical data reports, development of monitoring program, application preparation, and review of all project drawings.

Task Manager / Site Hydrogeologist
Phase II Investigation at a CERCLA site in New Jersey. Provided oversight and coordination of drilling, surveying, analytical and general services subcontractors. Activities involved soil sampling, test kit analysis, temporary piezometer installations via Geoprobe® techniques, monitoring well installation, aquifer testing, down hole video survey of site production well, well abandonment and demolition. All site activities were overseen and approved by a U.S. EPA oversight contractor.

Task Manager / Project Hydrogeologist
Soil and groundwater investigation at a RCRA Facility in West Virginia utilizing cone penetrometer deployed laser induced fluorescence for BTEX and PAH delineation. Additional activities included coordination land oversight of all subcontractors, oversight/implementation of all field activities including soil and groundwater sampling, groundwater monitoring, and aquifer testing.

Project Hydrogeologist
Pre-design investigation support for a former wood-treating site in Houston, Texas. Activities included installation of two six-inch DNAPL recovery wells, six piezometers and set-up and operation of a low flow long-term pumping test.

Project Engineering Geologist
For the design of a capping and shore protection project completed for the USACE at the Woodbridge Research Facility in Maryland.

Report Task Leader
For a groundwater investigation at a USACE Superfund Site on Long Island, NY. Activities included data evaluation, reporting, agency negotiation, and coordination of the deliverables.

Lead Project Geologist
For the completion of a RCRA closure at a Facility in Cleveland, Ohio. Activities included installation of several soil borings and sample collection. Following receipt of analytical data, performed statistical evaluation of data to closure criteria and assisted in report preparation.

Lead Project Geologist
Assisted in the preparation of a Part B Post-Closure Care Permit Application for closed RCRA surface impoundments at a RCRA Facility in West Virginia. Activities included extensive literature and regulatory review as well as report preparation.

RFI Field Investigation Task Leader
For a RCRA Facility in West Virginia. Activities included subsurface geophysical surveys to locate historical burial pits, subsequent oversight of several test pits, installation and operation of a temporary pneumatic product recovery well and sediment coring using a Vibra-Core sampler on an oxbow lake containing buried drums. Assisted in the preparation and completion of the RFI report.

Served as Task Manager/Hydrogeologist
For a Phase II Investigation at a CERCLA site in New Jersey. Provided oversight and coordination of drilling, surveying, analytical and general services subcontractors. Activities involved collection of over 200 soil samples and installation of twenty temporary piezometers using a Geoprobe®, monitoring well installation land subsequent sampling, aquifer testing, down hole video survey of site production well, well abandonment and demolition. All sampling activities were overseen and subsequently approved by a U.S. EPA oversight contractor.

Project Engineer
Assisted in the Remedial Design (RD) of a 22-acre CERCLA landfill located in a wetland that was tidally influenced and subject to flooding. RD activities included evaluating the stability of the cap by infinite slope stability analysis and evaluating the stability of the waste mass and underlying soils using the STABL5M computer software program developed at Purdue University. The RD approach proposed to the New Jersey Department of Environmental Protection (NJDEP) incorporates sediment barriers for sedimentation control as construction of traditional sedimentation basins was not feasible due to wetlands and flat Site topography. In addition, landfill side slope erosion protection in the form of erosion control matting was designed for protection from erosive flood waters and sheet flow over landfill side slopes.

Pre-Design investigation Field Support
For a former wood-treating site in Houston, Texas. Activities included installation of two six-inch DNAPL recovery wells, six piezometers and set-up and operation of a low flow long-term pumping test. Assisted in the Remedial Design of a treatment Facility in Houston, Texas. Activities involved pump sizing calculations for a groundwater extraction system, pipe failure analysis, and air line sizing/air loss calculations for a vapor extraction system.

Lead Field Activity Supervisor / Project Hydrogeologist
For a groundwater investigation at a chemical manufacturing facility in South Carolina. Activities included laboratory and subcontractor management and oversight, conduction of field activities, and preparation of the report. Approximately 25 Geoprobe® borings were installed and discrete soil and groundwater samples were collected to determine the extent of TCE

contamination. An on-site mobile laboratory was utilized to optimally locate monitoring wells following analysis of Geoprobe®-derived samples. In addition to the delineation activities, six temporary piezometers were installed to adequately determine groundwater flow direction and gradients in the investigative area. Also, a total of twenty monitoring wells were monitored for organics, metals, and field parameters to determine site-wide conditions. Based on the results obtained from these activities, a remedial evaluation was performed. The final report included an evaluation of data including microbial data obtained during the investigation for determination of active bioremediation. These data were later utilized in a Mixing Zone Application for the facility. Assisted in the preparation of a Mixing Zone Application for an active chemical manufacturing Facility in South Carolina. Activities included a review of over 10 years of previous analytical data reports, development of monitoring program, application preparation, and review of all project drawings.

Lead Project Hydrogeologist
For a DNAPL recovery program at a RCRA Facility in Ohio. Major activities included oversight of soil borings completed to obtain data on DNAPL extent. This data, along with data obtained through an extensive review of previous investigations at the Facility, was used to locate the recovery well. A thorough recovery well design as completed in an attempt to optimize the recovery of DNAPL. Following well installation, three recovery scenarios were implemented utilizing different pumping schemes to determine the best recovery setup. In addition to DNAPL recovery testing, intensive aquifer testing was completed to determine hydro-geological parameters. This information is currently being used to design a long-term recovery system.

Project Hydrogeologist
Assisted in the preparation of a Voluntary Cleanup Program Application for a former industrial facility in New York. Activities included a review of subsurface conditions as reported in previous reports, lead in addressing legal counsel and client comments to Application, and draft/final submittal activities.

Project Hydrogeologist
Performed data evaluation of resistivity surveys completed at a municipal landfill in Michigan. The purpose of the project was to determine the limits of waste in order to determine design limits for the cover system and surface water conveyance system. Also performed hydraulic calculations to determine surface water channel, retention pond, and cap designs for the landfill.

Publications

Coll, F. and Shakoor, A., Evaluation of Lime Dust as an Agent of Soil Stabilization for Soils and Waste Materials, Association of Engineering Geologists, Program and Abstracts, 37th Annual Meeting, Williamsburg, VA, October 1994.

Ruggery, D.A., Misquitta, N.J. and Coll, F.R., Delineation Of Creosote-Based Dnapls Using Cone Penetrometer-Deployed Laser Induced Fluorescence, presented at the Sixth Annual West Coast Conference on Contaminated Soils, Newport Beach, California, March 14, 1996.

Coll, F. and Shakoor, A., Physical and Chemical Characterization of Lime Dust as a Construction Material, Environmental and Engineering Geoscience, Summer 1996, v. II, No.2, pp. 183-193.

Coll, F.R., Hale, J.R. and Misquitta, N.J., Starting with the End in Mind: A Case Study Strategizing Data Collection During Phases of a Hydrogeologic Investigation, Abstracts With Programs, 1996 Annual Meeting, Geological Society of America, Denver, CO, October 28, 1996.

Coll, F., Misquitta, N., Brourman, M. and Foster, D., Innovative Well Design Techniques for Coal Tar DNAPL Recovery, National Groundwater Association 48th Annual Convention/Exposition, December 8-11, 1996, Las Vegas, Nevada.

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Standard Operating Procedures

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Quality Assurance Plan and General Procedures

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Date _____

LEGAL NOTICE

The FIRSTECHNOLOGY, Inc.'s Quality Assurance/Quality Control (QA/QC) Program and General Procedures is prepared from material available from Federal and State agencies and information obtained by the personnel of FIRSTECHNOLOGY, Inc. This document describes the basic methods implemented to analyze environmental samples and delineates FIRSTECHNOLOGY's adherence to Quality Assurance and Quality Control guidelines.

Although fully guaranteeing all results, the staff of FIRSTECHNOLOGY, Inc. makes no warranty, expressed or implied, concerning the accuracy, completeness or usefulness of the information contained *in this document*. FIRSTECHNOLOGY also assumes no liability with respect to the use of, or reliance upon any of the information provided in this document.

TABLE OF CONTENTS

	Page
1.0 **INTRODUCTION**	1
1.1 FIRSTECHNOLOGY's Organization and Responsibilities	
1.2 Assumed Client Responsibility and Disclaimer	
1.3 Current Certifications	
2.0 **LABORATORY ORGANIZATION**	5
2.1 Organization of Staff /Job Responsbilities	
2.2 Job Classifications - Education/Training Requirements	
2.3 Laboratory Design and Organization	
3.0 **TRAINING**	15
3.1 FIRSTECHNOLOGY Laboratory Orientation and Training Outline	
3.2 Training Records	
3.3 Competence of Operator, Procedure, and Matrix	
3.4 Commitment to Customers and Pride in Workmanship	
4.0 **MANUALS AND ANALYTICAL PROCEDURES**	19
4.1 Standard Operating Procedures and Document Control	
4.1.1. Procedures for Adoption and Revision of SOPS	
4.1.2. Revision of the Quality Assurance Manual	
4.1.3. Document Control Procedures	
4.2 Manuals as Sources of Information	
4.3 Metals Department SOPs - Outline	
4.4 Inorganics Department SOPs - Outline	
4.5 Organic Extractions Department SOPs - Outline	
4.6 Organic Analysis Department SOPs – Outline	
4.7 Quality Control Standard Operating Procedures – Outline	
4.8 Sample Control SOPs - Outline	
4.9 Personnel Policy Manual - Outline	
4.10 Chemical Hygiene Plan - Outline	
5.0 **LABORATORY PRACTICES**	27
5.1 Project Scheduling and Acceptance	
5.1.1. Subcontracted Work	
5.2 Sample Management during Collection / Shipment	
5.3 Handling Samples in the Laboratory	
5.4 Glassware Washing Procedures	
5.4.1 Special Cleaning Requirements	
5.5 Preparation, Purity and Storage of Reagents and Standards	
5.5.1 Laboratory Suppliers and Supplies	
5.6 Obtaining and Reporting of Detection Limits	
5.7 Miscellaneous Laboratory Practices	

TABLE OF CONTENTS
(Continued)

Page

6.0 **LIST OF MAJOR ANALYTICAL EQUIPMENT** 35
- 6.1 Organic Section
- 6.2 Metals Section
- 6.3 Wet Chemistry Section
- 6.4 Computer & Data Systems
- 6.5 Other

7.0 **INSTRUMENT CALIBRATION RECORDS** 39
- 7.1 Major Analytical Equipment
- 7.2 Support Equipment
- 7.3 Other Laboratory Equipment

8.0 **QUALITY CONTROL ANALYSIS** 42
- 8.1 Method Blank
- 8.2 Field Blanks/Trip Blanks
- 8.3 Reference Sample
- 8.4 Initial and Continuing Calibration Verification
- 8.5 Matrix Spike /Matrix Spike Duplicate
- 8.6 Surrogate Standard
- 8.7 Field Duplicates
- 8.8 Duplicate Data
- 8.9 Laboratory Control Samples
- 8.10 Standard Curves
- 8.11 Interelement Correction Factors
- 8.12 Contamination Control Samples
- 8.13 Corrective Actions

9.0 **METHOD GUIDELINE QC REQUIREMENTS** 47
- 9.1 Metals Analysis
  - 9.1.1. Drinking Water
  - 9.1.2. Liquid Analysis of Metals
  - 9.1.3. Solid Waste Analysis for Metals
  - 9.1.4. TCLP Analysis for Metals
- 9.2 Inorganic Analysis
  - 9.2.1. Drinking Water Inorganics
  - 9.2.2. Solid/Liquid Analysis of Inorganics
- 9.3 Organic Analysis
  - 9.3.1 Drinking Water THMs
  - 9.3.2 Drinking Water Volatiles

TABLE OF CONTENTS
(Continued)

Page

9.3.3 GC/MS Water/Wastewater Volatiles
9.3.4 GC Water/Wastewater Volatiles
9.3.5 GC/MS Water/Wastewater Extractables (BNA)
9.3.6 Drinking Water Pesticides/Herbicides
9.3.7 GC Water/Wastewater Pesticides

10.0 **STATISTICAL QUALITY CONTROL (SQC) & DOCUMENTATION** 53
10.1 Standard Curves
10.2 Matrix Spike (Known Addition) Percentage Recovery Calculations
10.3 Statistical Quality Control (SQC) Charts
10.4 Duplicate Analysis and Matrix Spike Determinations
10.5 Precision and Reproducibility

11.0 **DATA PROCESSING AND REPORTING** 57
11.1 Data Management, Documentation & Archiving
11.2 Documentation
11.2.1 Archiving/Back-up
11.2.2 Security
11.2.3 System Administration and Maintenance
11.3 Tracking of Samples and Analysis through the Laboratory
11.3.1 Login
11.3.2 Data Entry / Verification
11.3.3 Progress Reports
11.3.4 Analysis / Raw Data
11.3.5 Evaluation by Supervisor
11.3.6 Compilation/Generation
11.3.7 QC Checks
11.3.8 Typing / QC Proofreading
11.3.9 Data Qualifiers
11.3.10 Release of Analytical Report
11.3.11 Confidentiality of Reports
11.3.12 Final Written Reports
11.3.13 Billing
11.3.14 Filing
11.3.15 Sample, Chemical, and Solvent Disposal
11.4 Logbook Inventory
11.4.1 Bi-Weekly Inspections
11.4.2 Random Inspections

TABLE OF CONTENTS
(Continued)

	Page
12.0 **ADDITIONAL QUALITY ASSURANCE ITEMS**	64
12.1 Certifications	
12.2 External Audits	
12.2.1 New York DOH On Site Inspection	
12.2.2 Pennsylvania DEO On Site Inspection	
12.2.3 Ohio EPA On Site Inspections and Requirements	
12.3 Internal QC Audit	
12.4 Proficiency Evaluations	
12.5 Blind Samples	
12.6 Data Audits	
12.7 Inspections	
12.8 Irregularities/Corrective Action	
12.9 Report of Quality Assurance to Lab Management	
12.10 Drinking Water Contingency Plan	
12.11 Documentation of Customer Complaints	
13.0 **SAFETY AND THE CHEMICAL HYGIENE PLAN**	69
14.0 **SUMMARY**	70
15.0 **REFERENCES**	71

TABLE OF CONTENTS
(Continued)

APPENDICES

A Sample Label Example and Chain-of-Custody Form

B Environmental Sampling Guide

C Examples of Internal Analytical Result Report Forms

D Examples of Statistical Quality Control Charts

E Example of Final Analytical Report

F Methods and Method Detection Limits (MDLs)

1.0 INTRODUCTION

FIRSTECHNOLOGY, Inc. has prepared this quality assurance document to monitor, control and establish the factors which contribute to the validity of analytical results. FIRSTECHNOLOGY assumes the responsibility to adhere and implement EPA-approved protocols to assure the scientific reliability of its analytical data. FIRSTECHNOLOGY, currently housed in a 10,000 square-foot building, is a full-service environmental analytical laboratory. Our analytical capabilities include organic, metals, inorganic and other miscellaneous analysis covering a wide range of parameters. FIRSTECHNOLOGY is equipped with the leading edge of analytical equipment and has a well-qualified technical staff of chemists and technicians (Figure 1) to insure strict adherence to quality assurance (QA) and quality control (QC).

1.0.1 FIRSTECHNOLOGY's Quality System Policies and Objectives

The objective of the FIRSTECHNOLOGY laboratory is to provide information that is factual, accurate, precise, reliable and adequate for our customers' use. The QA and QC procedures are used to ensure the achievement of the program objectives and QA is the system for ensuring that all information, data, and resulting decisions compiled under specific tasks are technically sound, statistically valid and properly documented. Quality control is the mechanism through which quality assurance achieves its goal. Quality control programs define the frequency and method of checks, audits and reviews necessary to identify problems and dictate corrective action, thus verifying product quality (Figure 2).

1.1 FIRSTECHNOLOGY's Organization and Responsibilities

FIRSTECHNOLOGY's environmental laboratory is responsible for the analysis of all samples provided, including complete QA/QC results for each individual analyte.

The laboratory management staff of FIRSTECHNOLOGY is committed to following the standards outlined in this document in order to maintain the highest standard of quality.

Laboratory management will ensure that all personnel hired possess the appropriate education and/or technical background for the position they will be assuming. (See Sec 2.2 for lab positions and educational requirements).

Laboratory management is responsible for making sure all employees are free from any pressures-commercial, financial, or other that might influence their judgments in making technical decisions with regards to laboratory data. No employee is offered financial bonuses as incentive to hasten work, or coerced into compromising quality control data. Laboratory management has made clear to employees that intentionally compromising the quality standards of FIRSTECHNOLOGY is grounds for termination of employment. Management also ensures that employees hired are free from conflicts of interest that could arise in performing their duties as an employee of FIRSTECHNOLOGY.

All personnel are required to be familiar with the latest revision of this Quality Assurance Plan, as well as the latest revisions of relevant test methods. All personnel are required to implement the applicable policies and procedures defined within this document in their daily work.

1.2 Assumed Client Responsibility and Disclaimer

FIRSTECHNOLOGY, Inc. shall provide the services contained in accordance with good laboratory practice (GLP), and accepted analytical procedures and shall be free from material defect in workmanship. The analytical data is limited to findings based upon the sample received for analysis and/or information provided by the client. FIRSTECHNOLOGY's sole obligation hereunder shall be to reperform services which are materially deficient because of FIRSTECHNOLOGY's failure to perform said services in accordance with this Agreement and the standards of the laboratory analytical protocol. Any such deficiencies should be reported in writing to FIRSTECHNOLOGY within thirty days of the discovery thereof, but in no event later than one year from the performance of the services by FIRSTECHNOLOGY.

Except as aforementioned, FIRSTECHNOLOGY makes no express or implied warranty of merchantability or fitness for a particular purpose on the services and/or related materials furnished by FIRSTECHNOLOGY. In no event shall FIRSTECHNOLOGY be liable for any indirect, special or consequential damages, nor shall FIRSTECHNOLOGY be liable in any event, including its obligation to reperform, for any losses, damages or claims in excess of the amount paid to FIRSTECHNOLOGY for the services performed.

1.3 Current Certifications

FIRSTECHNOLOGY's quality of results is currently certified in the analysis of wastewater and solid waste by the New York State Department of Health ELAP program. Also, FIRSTECHNOLOGY is certified for drinking water by the Ohio EPA. Maintenance of these certifications requires periodic on-site audits and analysis of performance evaluation samples, at least semi-annually, prepared by the respective agencies or acquired from an approved source.

1.3.1 NELAC Quality System Requirements

FIRSTECHNOLOGY has established its' quality assurance plan to be in conformance to all NELAC Quality System requirements as established in NELAC Chapter 5, July 2002.

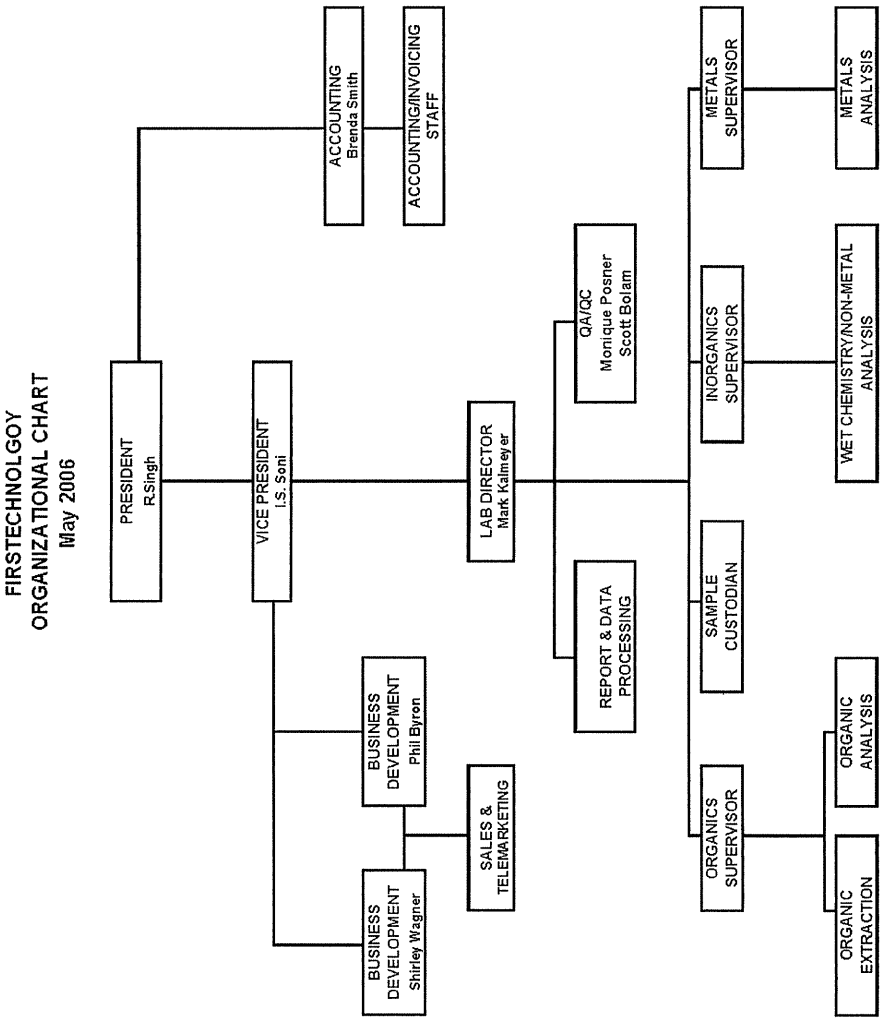
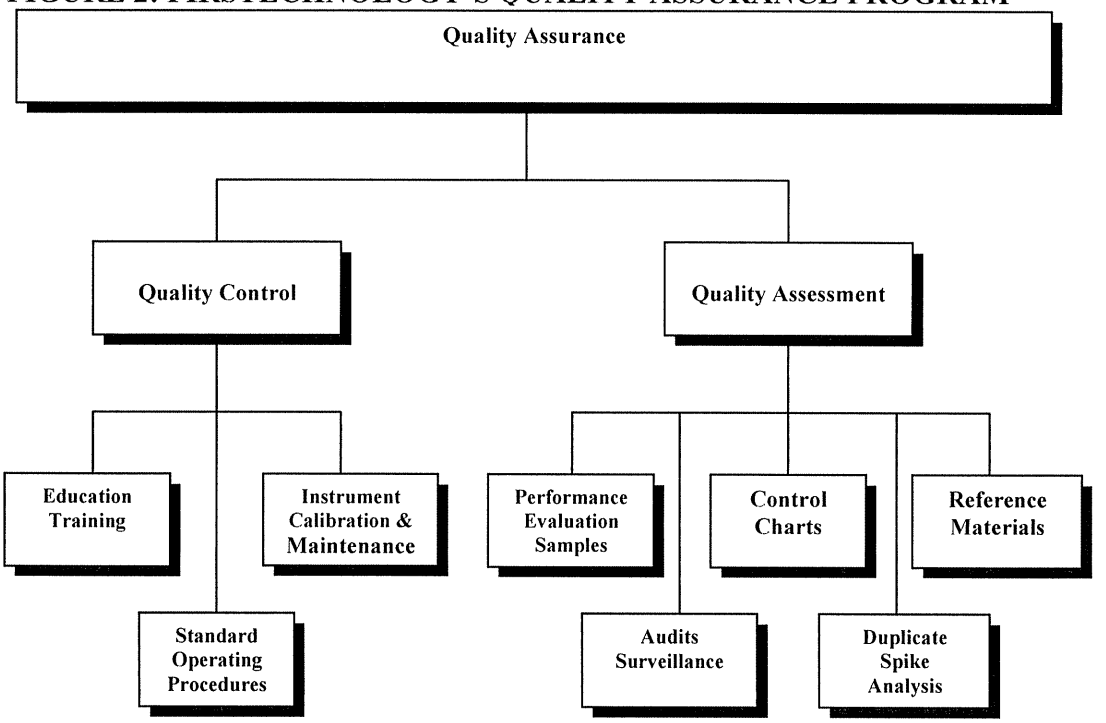


FIGURE 1. THE FIRSTECHNOLOGY LABORATORY ORGANIZATION

FIGURE 2: FIRSTECHNOLOGY'S QUALITY ASSURANCE PROGRAM



Basic components of the laboratory quality control

Trained and qualified analysts and support personnel
Standard Operating Procedures (SOPs)
Instrument calibration and maintenance system
Reliable instruments and equipment
Documentation of analytical and operational information
Proper sampling and sample tracking techniques
Appropriate methodology usage
Good laboratory practices

Basic components of the laboratory quality assessment

Performance evaluation samples
Audits and surveillances
Duplicates, spikes, surrogates, blind analyses
Standard reference materials
Control charts
Reference materials
Round-robin collaborative testing
Statistical analysis of data
Corrective actions

2.0 LABORATORY ORGANIZATION

FIRSTECHNOLOGY has taken great care in organizing its departments and the job responsibilities of people within each department to provide customers with a continuous throughput of information through the organization levels as well as through the various departments of the company.

2.1 Organization of Staff/ Job Responsibilities

The following job responsibilities represent the chain of command and supervision of personnel at FIRSTECHNOLOGY, Inc.

Laboratory Director:

- Mandates that a high degree of quality in the analytical results is maintained and reported in a timely manner.
- Designs and maintains the paperwork system of the laboratory.
- Plans improvements in procedures, instrumentation and personnel.
- Reviews QA/QC reports.
- Develops the operating budget for the laboratory.
- Assures that a high degree of quality in the analytical results is maintained and reported in a timely manner.
- Reviews QA/QC reports.
- Directs and guides section supervisors.
- Authorizes the purchase of requested supplies.
- Assesses new work and projects to ensure that appropriate facilities and resources are available before commencing such work.
- Assumes backup duties for the QA/QC Officer
- In the event that the Laboratory Director is not available, a deputy will assume responsibilities of the Laboratory Director. Typically this is the Quality Assurance Manager.

Quality Assurance Manager:

- Sees that all results reported from the laboratory are reviewed for precision, accuracy and completeness.
- Prepares and maintains quality control graphs and charts.
- Conducts quality assurance audits of the laboratory.

- Plans and implements improvements in laboratory procedures to control the reliability of data. Also takes corrective action in instances where protocols are out-of-control.
- Performs other tasks as assigned.
- Assume backup duties for the Laboratory Director.
- In the event that the QA/QC Manager is unavailable, a deputy will assume responsibilities of the QA/QC Manager. This is typically the Lab Manager or other supervisory position.

Report Coordinator:

- Coordinates sample log in procedures to maintain efficient laboratory throughput.
- Tracks and compiles results from respective analytical sections.
- Reviews results and prepares reports for customers.
- Performs other tasks as assigned by the Laboratory Manager.

Supervisor of Organic Analysis:

- Oversees the preparation and analysis of organic constituents.
- Plans time and effort to improve the throughput and efficiency in the organic section.
- Provides the QA Manager with the appropriate data for preparation of the quality control charts of all organic analyses.
- Suggests and implements improvements in organic analytical procedures and insures preventive maintenance of associate equipment.
- Orders replacement supplies for the organic section.
- Performs other tasks as assigned by the Laboratory Manager.

Supervisor of Metals analytical section:

- Oversees the preparation and analysis of metal constituents.
- Plans time and effort to improve the throughput and efficiency in the metals section.
- Provides the QA Manager with the appropriate data for preparation of the quality control charts of all metal analyses.
- Suggests and implements improvements in metals analytical procedures and insures preventive maintenance of all associated equipment.

- Orders replacement supplies for the metals section.
- Performs other tasks as assigned by the Laboratory Manager.

Supervisor of Inorganic analysis section:

- Oversees the preparation and analysis of inorganic constituents.
- Plans time and effort to improve or maintain throughput an efficiency in the inorganic section.
- Provides the QA Manager with the appropriate data for preparation of the quality control charts of all inorganic analyses.
- Suggests and implements improvements in inorganic analytical procedures and insures preventive maintenance of associated equipment.
- Orders supplies for the inorganic section.
- Performs other tasks as assigned by the Laboratory Manager.

Sample Custodian:

- Logs in all samples coming into the laboratory.
- Inspects samples for correct storage conditions and takes corrective action if necessary.
- Sees that Chain-of-custody forms are filled out correctly and forwarded to data entry personnel for entering into the laboratory sample tracking system. He/she also makes notations should the samples not comply with sampling protocol.
- Tracks all samples from initial reception to disposal and can determine their whereabouts at all times.
- Keeps a daily temperature record of refrigerators used for sample storage and initiates corrective actions when this storage equipment deviates from the correct temperature. Also records the conductivity of reagent-grade water on a daily basis and analytical balance calibration on a weekly basis.
- Keeps an accurate inventory and adequate stock of clean sample containers and, when required, prepares the appropriate containers with the correct preservative for use by customers of FIRSTECHNOLOGY.
- Performs other tasks as assigned by the Laboratory Manager.

Other Employees:

- Operate analytical equipment.
- Prepare samples, standards and reagents.

- Complete all paper work in a timely manner.
- Maintain the laboratory as a clean and safe work place.
- Notify their supervisors of any irregularities in the procedures for which they are responsible.
- Perform other tasks as assigned by the Laboratory Manager or section supervisor.

2.2 Job Classifications - Education/Training Requirements

In addition to job responsibilities, there are educational and professional experiences levels which provide classifications for the type of supervision required for daily work.

Inorganics

- **Technician Level I**
  An individual with no/minimal experience in a general lab environment. Extensive training required.
  Education: High School diploma.

- **Technician Level II**
  An individual with over one year experience in inorganic environmental analysis, knowledge of specific types of inorganic analysis, minimum training requirements.
  Education: high school diploma.

  An individual with a college degree in chemistry of related science and less than a year in general lab experience.
  Education: Associates degree in science field.

- **Chemist Level I**
  An individual with two years experience in inorganic environmental analysis, requiring minimal supervision in all areas of inorganic analysis, knowledgeable in EPA protocol.
  Education: Bachelors degree in science field.

- **Chemist Level II**
  Individual with four years experience in inorganic environmental analysis, knowledgeable in all areas of inorganic analysis and completely familiar with EPA protocol. Requires no supervision.
  Education: Bachelors degree in science field.

- **Supervisor Level I**
  Individual with minimum three years environmental lab experience, knowledge of most inorganic procedures and completely familiar with EPA protocol, minimum training required. Capable of trouble shooting any wet chemistry related problems.
  Education: Bachelors degree in science field.

- **Supervisor Level II**
  An individual with a minimum of three years inorganic experience, knowledgeable in all inorganic parameters and EPA protocol. Works independently.
  Education: Bachelors degree in science field.

Organic Extractions

- **Technician Level I**
  An individual with no/minimal experience in a general lab environment. Extensive training required.
  Education: High school diploma.

- **Technician Level II**
  An individual with over six months experience in organic extractions, knowledge of specific types of extractions, minimum training required.
  Education: High school diploma.

  An individual with less than two years lab experience, minimum training required.
  Education: Bachelors degree in science field.

- **Technician Level III**
  An individual with over two years experience in organic extractions, knowledgeable with all extraction techniques and EPA protocol, problem solving ability regarding extractions, minimal supervision required.
  Education: Bachelors degree in science field.

Organic Analysis

- **Technician Level I**
  An individual with no/minimal experience involving GC; GC/MS instrumental analysis, extensive training required.
  Education: Associates degree in science field.

- **Technician Level II**
  An individual with over two years experience in GC; GC/MS analysis, capable of running samples, surrogate and matrix spiking, limited data interpretation abilities.
  Education: Associates degree in science field.

  An individual with one year of experience in GC; GC/MS.
  Education: Bachelors degree in science field.

- **Chemist Level I**
  An individual with two years of hands on experience doing GC or GC/MS analysis, requires minimal supervision in all areas of analysis, knowledgeable of EPA protocol, performs routing maintenance on instrument, possesses trouble shooting capabilities, requires data validation by group supervisor, experience in extraction and clean up procedures.
  Education: Associates degree in science field.

- **Chemist Level II**
  An individual with four years of hands-on experience doing GC and GC/MS analysis, knowledgeable in all areas of analysis and EPA protocol, requires no supervision, capable of routing maintenance and troubleshooting instrumentation, experience with all necessary extractions and clean-up procedures.
  Education: Bachelors degree in science field.

- **Supervisor Level I**
  Minimum of five years GC; GC/MS experience, knowledge of all extraction techniques and EPA protocol, capable of utilizing all analytical instrumentation, trouble shooting and maintenance capabilities, no training required.
  Education: Bachelors degree in science field.

- **Supervisor Level II**
  An individual with a minimum of seven years experience in GC; GC/MS analysis, knowledgeable of all organic parameters and EPA protocol. Responsible for all activities involving the organic group. Fully independent, no training/supervision required.
  Education: Bachelors degree in science field.

Metals

- **Technician Level I**
  An individual with no/minimal experience in a general lab environment. Extensive training required.
  Education: High school diploma.

- **Technician Level II**
  An individual with over one year experience in sample preparation, knowledge of specific types of digestion procedures, minimum training requirements.
  Education: High school diploma.

  An individual with three years of lab experience with one of those years involving sample preparation for metals analysis.
  Education: High school diploma.

  An individual with less than one year of general lab experience.
  Education: Associates degree in science field.

- **Chemist Level I**
  An individual with two years experience doing metals preparation and analysis. Requires minimal supervision in all areas of metals preparation and analysis. Knowledgeable of EPA protocol, familiar with all instrumentation in metals department. Performs routine maintenance but requires data validation by supervisor.
  Education: Bachelors degree in science field.

- **Chemist Level II**
  An individual with four years experience in metals preparation and analysis. Knowledgeable in all areas of metals analysis and familiar with EPA protocol, requires no supervision, capable of maintaining and troubleshooting all metals instruments.
  Education: Bachelors in science field.

- **Supervisor Level I**
  An individual with five years metals experience, knowledgeable of metals analytical procedures and EPA protocols, capable of utilizing all metals instrumentation, trouble shooting and instrument maintenance capabilities. Requires minimal training and works independently.
  Education: Bachelors degree in science field.

- **Supervisor Level II**
  An individual with five years metals preparation and analysis, knowledgeable of all metals parameters and EPA protocols. Responsible for all activities involving the metals analysis. Fully independent.
  Education: Bachelors degree in science.

Sample Custodian

- **Level I**
  An individual with minimal/no experience in sample processing procedures, minimal lab experience. Extensive training required.
  Education: High school diploma.

- **Level II**
  An individual capable of fulfilling all the job requirements of the sample custodian.
  Education: Associates degree.

Report Generation

- **Typist I**
  An individual with minimal/no experience in technical, clerical, and computer skills. Extensive training required.
  Education: High school diploma.

- **Typist II**
  An individual with a minimum of two years technical clerical and computer experience, requires little supervision. Individual has other responsibilities involving office functions (ordering supplies, maintaining computer system, filing reports, etc.), data entry and other duties as assigned.
  Education: High school diploma.

- **Analytical Data Coordinator**
  An individual responsible for the generation of progress report sheets, proofing chains of custody, compiling data from analysts for reporting, formation of initial reports, working with management in the prioritizing of work, tracks data from respective analytical sections and performs other tasks as assigned by management.
  Education: Bachelors degree in science field.

Quality Control

- **Quality Control Officer**
  An individual responsible for implementation and improvement of the laboratory quality control procedures. Responsible for maintaining lab certification from various agencies. Individual must be a self-starter, require minimal or no supervision, be knowledgeable in the utilization of spread sheets and work processing. Perform other duties as assigned by management.
  Education: Bachelors degree in science field.

Management

- **Laboratory Director**
  An individual with at least two years of experience in a water laboratory who is responsible for all laboratory activities including equipping the lab, maintaining certification, interviewing and hiring technical personnel with expertise in the environmental field. Implement ideas to augment analytical capabilities and to promote business growth. Motivate and encourage personnel to increase efficiency and productivity with great emphasis on quality and service. Maintains network of communication with regulatory agencies. Actively involved with the management of business in decision making and other financial matters to enhance the productivity of the lab operation. Familiar with various phases of hazardous waste management.
  Education: Bachelor of Science or Arts degree in a scientific field.

- **Laboratory Manager**
  An individual with at least one year of experience in the chemical analysis of water. Responsible for all aspects of laboratory operations including supervision of all lab personnel. Planning and implementation of analytical procedures as per USEPA protocol. Assist sales people to provide technical support and other environmental services to the customer. Actively involved in business decisions, hiring of personnel and purchasing equipment. Manage budget in various departments and organize personnel to maximize the throughput in the lab. Check analytical data, computer printouts and timely release of analytical reports to the customer on a daily basis.
  Education: An individual with a bachelor's degree in a science field that includes chemistry courses.

2.3 Plan for Going out of Business / Transfer of Ownership

In the event that FIRSTECHNOLOGY is purchased and transferred to new ownership, all clients will be notified of the pending change of ownership. The current ownership of FIRSTECHNOLOGY will determine if customer records will continue to be maintained by the new ownership. If not, arrangements will be made, at the expense of customers, for them to take receipt of existing records stored by FIRSTECHNOLOGY.

In the event of a close of business scenario, all clients will be notified of the pending closure. Clients will be offered to take possession of their existing records, at the client's expense. Clients will be informed of any regulatory limitations on time of storage of records.

2.4 Laboratory Design and Organization

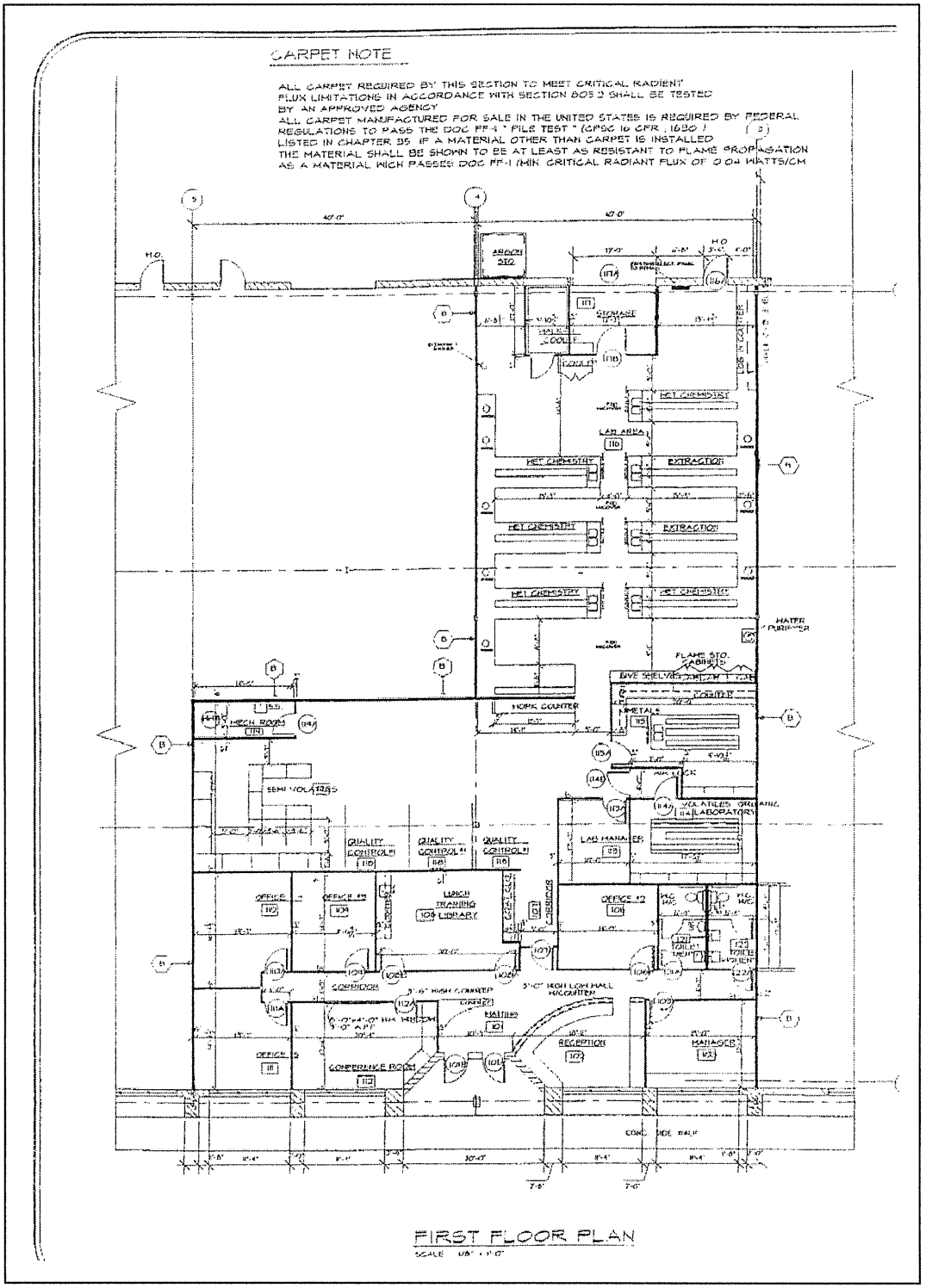
In order to reduce the probability of cross contamination the FIRSTECHNOLOGY laboratory is divided into three (3) major groups:

1) Organic Group
2) Metals Group
3) Wet chemistry (inorganic) group

Each group or section has a designated preparation area in addition to its own glassware, washing station, drying racks, drying ovens, etc. Furthermore, the laboratory also has a designated room utilized exclusively for cleaning contaminated glassware generated by the various analytical groups.

The following diagram (Figure 3) shows a floor plan of the laboratory area of FIRSTECHNOLOGY, Inc.

FIGURE 3: THE FIRSTECHNOLOGY LABORATORY FLOOR PLAN



3.0 TRAINING

3.1 FIRSTECHNOLOGY Laboratory Orientation and Training Outline

Laboratory Tour, General

Wet chemistry group
Metals group
Organics group
Sample login and storage
Waste accumulation area
Glassware washing room

Laboratory Tour, Health & Safety

Fire extinguishers
Fire exits
Fire blankets
Safety deluge showers and eyewash stations
First-aid cabinets
Chemical storage cabinets
Location of MSDS books
Safety eyeglass areas

Health & Safety Policies and Procedures

Read Chemical Hygiene Plan
No smoking policy
Eating area
Use of safety eyeglasses, lab. coats, gloves
Dress code
Hand washing
View safety training video tape
Take safety test
Know initiation of emergency response system (9-1-1)

Read Quality Assurance Plan

Discuss (with QAO) and understand:
Quality control concepts, definitions, requirements and calculations, blanks, standards, linearity, ICV, LCS, MS, surrogates, % recovery, duplicates
Documentation policies & procedures
Logbooks and test sheets
Sign Pledge of Quality and Commitment
Discuss and understand ethical and legal responsibility SOP, sign Agreement of Ethics; review annually

Specific Method Training

Read and understand published method and FIRSTECHNOLOGY SOP
Preceptor (teacher, group leader) hands-on training through entire sample preparation and analysis
Operator Competence

Miscellaneous

Glassware washing
Joblist assignment sheet (or progress report)
Supply ordering policy
Records of Training

3.2 Training Records

Records of on the job training, seminars attended, and safety meetings attended are recorded in a general training logbook kept with the Quality Assurance Officer and the Lab Manager.

This logbook contains the date, a description of the training, and who was the person who performed the instruction. Each employee is requested to notify the QA Officer or the Lab Manager of any seminar or continuing education activity performed outside of work that directly relates to the work environment. By doing so, a record of your experience and qualifications can be maintained.

All continuing demonstrations of proficiency are also kept as part of employee training records. Also, any annual review documentation is kept updated and in the employee training records.

The employee training records also contain any documentation as required by specific certification agencies. These requirements are found in each certification program's documentation.

3.3. Competence of Operator, Procedure, and Matrix

When a new analyst begins to work in the laboratory, he/she works under the direct supervision of the immediate supervisor. During this time the new analyst should demonstrate competence of the testing method. The following recommendation may be used to show competence in a testing procedure. It is also recommended that this procedure be used when a new method is being used for the first time in the laboratory and when a new matrix type is being analyzed using an established method.

Competence of Operator
Operator must make four replicates of an independently prepared check sample with a concentration between 5 and 50 times base DL and recover within the below listed ranges.

Metals	80% - 120%
Volatile	70% - 130%
Base Neutrals	70% - 130%
Acids	60% - 140%
Inorganics	80% - 120%
Herbicides	40% - 160%
Pesticides	50% - 140%

Competence of Matrix (Recovery of Known Additions)
Known additions must be used to verify the absence of matrix effects. When a new matrix is analyzed, verify the amount of interference.

Spikes must represent 10% of the samples and should be made at a value between 5 and 50 times the base detection limit. Duplicates should also be performed on 10% of the samples.

Competence of Instrumentation (Analysis of Standards)
A laboratory control standard should be analyzed once each day and should be at a concentration between 5 and 50 times the base detection limit. These standards should be prepared independently of standards used for calibration.

Competence of Instrumentation (Analysis of reagent blank)
A minimum of 1 in 20 samples should be reagent blanks. A reagent blank should be analyzed after any sample with a concentration greater than that of the highest standard that might result in carryover from one sample to the next.

Competence of Quantification (Calibration with standards)
Calibration should be made with a blank and at least three standards and verified using at least one standard within the linear range. Results for samples should not be reported above the highest standard unless an initial demonstration of a greater linear range has been made and no instrument parameters have been changed since. In addition the value must be less than 1.5 times the highest standard.

Competence of Sampling Procedures (Analysis of duplicates)
Analysis for duplicates must be at least one in twenty samples, some procedures including metals and inorganic analysis requires a one in ten duplicate ratio.

3.4 Commitment to Customers and Pride in Workmanship

Confidentiality of Results
All results obtained from samples from any client are strictly confidential and will not be released to anyone except the client unless a written request by the customer has been made. To assure and conform with this policy all requests for analytical information are to be addressed to the laboratory director or a designated authorized representative. When records are to be disposed of, all records containing specific client information will be shredded or destroyed such that no client information can be reconstructed.

Commitment to Training and Education
Each employee at FIRSTECHNOLOGY has pledged to follow the QA/QC protocol pertaining to the particular methodology being employed, including but not limited to matrix spikes, duplicates, method blanks, etc.

Each employee is familiar with the methodology prior to utilizing the method or being trained for the utilization of the method. This is accomplished by reading all relevant material pertaining to the said methodology including the Standard Operating Procedure, EPA methodology (both water/waste water method and solid waste method) and the Standard Method if applicable and working under the direct supervision of the group supervisor until the employee has demonstrated the capacity to work independently. Each employee is kept up-to-date on the latest revisions of Quality Documentation and applicable SOPs.

Commitment to Service
Each employee is dedicated to utilize his/her time efficiently, for the sole purpose of producing analytical results within the specified turn-around time, in order to avoid any delays in the reporting of the analysis to the customer. Also each employee will assist people in another group after completion of their own work.

Pride in Workmanship
Each employee assumes responsibility for the work that he/she produces, and states that is will meet or exceed all the QA/QC requirements pertaining to the method utilized and to see that all reagent and supplies used are maintained as to prevent any potential delay in analysis.

Employee Ethical and Legal Responsibilities/Pledge of Quality, Commitment and Ethics

Each employee working in the laboratory has been instructed in the required ethical standards of behavior for FIRSTECHNOLOGY, as outlined in *SOP/QC/4000: Data Integrity and Ethical Behavior.* Employees are given a summary list of ethics required by FIRSTECHNOLOGY, as well as examples of improper behavior, and have been made aware that not adhering to such standards could result in termination, or civil/criminal prosecution. Finally, each employee and the management personnel providing the ethical training are required to sign an Agreement of Ethics. These Agreements are kept in the employee personnel file. Ethical behavior will be reviewed at least once a year for all employees. Employees will sign a new Agreement of Ethics each year after ethical behavior has been reviewed. More detailed ethical training may be provided if necessary. A person of management is designated as a data integrity officer, through which employee concerns regarding ethical issues can be handled.

Each employee has signed a Pledge of Quality and Commitment, agreeing to meet or exceed all QA/QC protocols the FIRSTECHNOLOGY Quality System. If proper QA/QC guidelines are not met, the analyst will stop all work until all QA/QC issues are satisfactorily resolved and all QA/QC requirements are in full compliance.

Each employee has agreed to inform management, in a timely manner, of any potential problems regarding analysis, lab supplies, health/safety concerns or any other issues that will affect the productivity and welfare of the employee or the company.

Evaluation of data integrity is done quarterly using notebook review (see section 12.6). The QA/QC department reviews all laboratory notebooks quarterly, logs the results, and notes any deficiencies or discrepancies. Any findings that may be violations of the Data Integrity and Ethics policy are reported to the data integrity officer and management for further action. In addition, the QA/QC department will perform a full audit on 7-10 sample packages to determine if all proper documentation and traceability is being maintained from sample receiving to final reporting. These findings are also compiled and reviewed at the end of the year during the annual report to management. Serious violations of policy can result in termination of employment or legal action.

4.0 MANUALS AND SOURCES OF INFORMATION

4.1 Standard Operating Procedures (SOPs) and Lab Documents

Standard Operating Procedures exist for all procedures in the lab. The methods in use at FIRSTECHNOLOGY, Inc. represent methods in the form of standard operating procedures and represent the application of the approved EPA methodology to our laboratory. Each Standard Operating Procedure contains an analytical method reference to any of the following methods types:

- Water/wastewater methods
- Solid waste methods
- Standard methods
- ASTM methods

Standard Operating Procedures are available to all personnel. These documents are updated and revised on a regular basis and reviewed annually for content, compliance and relevance. The Quality Control officer maintains a record of all document revisions and updates, as well as the distribution of controlled copies. All personnel are required to be familiar with the most up-to-date revisions of procedures that they are using. Records are maintained documenting which controlled revisions an analyst is using.

Implemented standard operating procedures are such procedures that are signed by the lab manager and QA/QC officer and indicate the Effective Date, Revision Number, SOP ID, Title, and approval signatures on the front cover. The header of the following pages includes the SOP ID, Title, Revision Number, and Effective Date. Each method SOP should contain the following information:

- Analyte name with qualifier
- Approved methods of reference
- Applicable Matrices
- Detection Limit
- Scope and Application
- Summary of method
- Definitions
- Known interferences
- Environmental Health & Safety Protocols
- Equipment and Supplies
- Reagents and Standards
- Sample Handling and Preservation
- Quality Control Requirements
- Calibration and Standardization Procedures
- Analytical Procedure
- Calculations
- Method Performance
- Data Assessment and Corrective Action Procedures
- Contingencies for Out-of-Control Data
- Waste management and Pollution Prevention
- References

In all situations, a developed SOP should reflect what is currently practiced and provides a training tool for new personnel. It assures that the method is totally followed.

For distribution to employees, a control block appears on the front cover indicating the copy number, the date issued, and the initials of the person issuing the document. Official controlled copies are printed on pink paper to easily identify their status. This information is recorded by the QA/QC department to track the location and status of all controlled copies.

4.1.1 Procedures for Adoption and Revision of SOPs

Standard operating procedures are developed by the department supervisor in conjunction with the QA officer. Once the method is presented in the correct format, it is reviewed by the QA officer and the department supervisor for content and compliance to the method requirements. The final draft is presented to the lab manager, who will sign and date the front cover after final approval.

Changes and/or corrections are handled in a similar manner. A change to a standard operating procedure must be brought to the attention of the QA/QC officer with a reason for the change and then presented to the lab manager for approval.

Corrections and helpful notes to a SOP should not exist in a handwritten form in a working copy of an SOP. If these notes are important they should be incorporated into the SOP or into the analysis logbook. Otherwise all handwritten messages in an SOP should be removed.

Changes to be made prior to issuing a corrected, updated revision can be handwritten in as long as QA/QC sign and date the changes as a sign of approval. These revisions should be incorporated into a final, new revision as soon as possible.

All standard operating procedures, the Quality Assurance Manual, the Chemical Hygiene Plan and this manual are reviewed at least annually for content and potential revisions.

Each page of the SOP will contain the effective date, revision number, document number, and document title. As a controlled document, there must be an approval signature page, a record of revision history, and a record of distribution.

4.1.2 Revision of the Quality Assurance Manual

At minimum on an annual basis, the Quality Assurance Manual will be evaluated for content, relevance, and correctness. The Quality Assurance Manual should also be reviewed following major operational changes within the laboratory. Any changes to procedures that affect the quality assurance manual will be evaluated to verify compliance with the new methodology requirements. The final draft is presented to the lab director and QA/QC officer, each of which must approve the Quality Assurance Manual by signing and dating the front cover.

4.1.3 Document Control Procedures

Document Control in the analytical lab is needed to ensure that personnel have current methods, specifications, and standards in their possession and those employees are not using obsolete documents. A document control system ensures that the analyses and calibrations are carried out in accordance with the latest analytical documents. It also allows for an effective communication system, a centralized storage area, and an issue point for documents and changes in methodologies which permits legal and accreditation requirements to be met.

All operating procedures and documents are considered controlled documents, and subject to document control. Controlled documents are indicated by the proper paper color listed in the footer. Uncontrolled copies are indicated by reproduction on any other type of paper. The Quality Control officer maintains a record of all controlled copies of documents, and their location. Obsolete documents may be retained for legal reasons or knowledge preservation. All SOP, Quality Assurance Manual, and Chemical Hygiene Plan revisions are archived to allow the lab to historically trace the methods used for samples analyzed. The Quality Control officer retains all obsolete documents that need to be kept.

A Microsoft Access database is used by the Quality Control officer to track document revision history and control copy distribution.

A controlled document will contain the effective date, revision number, document number, and document title on each page. There must be an approval signature page for each SOP. Controlled forms are initialed in the header.

4.2 Manuals as Sources of Information

The Manuals and Standard Operating Procedures at FIRSTECHNOLOGY are the major sources of information regarding procedures and policies that are implemented at this company. These manuals and procedures are available to all personnel.

The following pages provide a general outline of content for each of the standard operating procedures present at FIRSTECHNOLOGY.

4.3 Metals Department Standard Operating Procedures - Outline

SOP/M/1000	Metals Sample Preparation
SOP/M/1050	Washing Glassware for Metals Analysis
SOP/M/1100	Wipe Sampling of Laboratory Surfaces
SOP/M/1500	Metals Analysis by Inductively Coupled Plasma Emission Spectrometry (ICP-ES)
SOP/M/1600	Mercury by Manual Cold Vapor Atomic Absorption
SOP/M/1700	Metals by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)
SOP/M/1800	Metals Analysis by Graphite Furnace Atomic Absorption (GFAA)

4.4 Inorganics Department Standard Operating Procedures - Outline

SOP/WC/1000	Acidity
SOP/WC/1010	Alkalinity
SOP/WC/1020	Biochemical Oxygen Demand (BOD) / (CBOD)
SOP/WC/1030	Bromide
SOP/WC/1050	Caloric Content (BTU)
SOP/WC/1060	COD
SOP/WC/1070	Chloride
SOP/WC/1090	Chlorine, residual
SOP/WC/1100	Chlorine by bomb
SOP/WC/1110	Chromium, Hexavalent
SOP/WC/1120	Color
SOP/WC/1130	Conductance
SOP/WC/1140	Cyanide, Free
SOP/WC/1150	Cyanide, Reactive
SOP/WC/1160	Cyanide, Total
SOP/WC/1170	Cyanide, Amenable to Chlorination
SOP/WC/1180	Fluoride
SOP/WC/1190	Formaldehyde
SOP/WC/1200	Ignitability/Flashpoint
SOP/WC/1210	MBAS, Surfactants
SOP/WC/1220	Nitrogen, Ammonia
SOP/WC/1230	Nitrogen, Kjeldahl
SOP/WC/1240	Nitrogen, Nitrate/NO3 + NO2
SOP/WC/1250	Nitrogen, Nitrite
SOP/WC/1260	Oil & Grease/FTIR
SOP/WC/1270	Oil & Grease/Gravimetric
SOP/WC/1280	Oxygen, Dissolved
SOP/WC/1290	TPH, Total Recoverable
SOP/WC/1310	Total Organic Halides (TOX)
SOP/WC/1320	Paint Filter Test
SOP/WC/1330	pH
SOP/WC/1340	pH, Soil
SOP/WC/1350	Phenolics
SOP/WC/1360	Phosphorous/All Forms
SOP/WC/1370	Total Solids
SOP/WC/1380	Total Suspended Solids
SOP/WC/1390	Total Volatile Solids
SOP/WC/1400	Total Dissolved Solids
SOP/WC/1410	Total Settleable Solids
SOP/WC/1420	Specific Gravity
SOP/WC/1430	Sulfite
SOP/WC/1440	Sulfate
SOP/WC/1450	Sulfides, Total
SOP/WC/1460	Sulfides, Reactive

SOP/WC/1470	TCLP Extraction
SOP/WC/1480	Turbidity
SOP/WC/1490	Total Organic Carbon (TOC)
SOP/WC/1500	LACHAT General Procedures

4.5 Organic Extractions Department Standard Operating Procedures - Outline

SOP/OE/1500	Organic Extractions General
SOP/OE/1600	Semivolatile Liquid/Liquid
SOP/OE/1700	Semivolatile by Sonication
SOP/OE/1800	Semivolatile, Pest, PCB by Waste Dilution
SOP/OE/1900	Pest/PCB Liquid/Liquid
SOP/OE/2000	Pest/PCB by Sonication
SOP/OE/2100	Herbicides Liquid/Liquid
SOP/OE/2200	Herbicides by Sonication
SOP/OE/2300	Herbicides by Waste Diltuion
SOP/OE/2400	Pesticide Clean-Up
SOP/OE/2500	PCB Clean-Up
SOP/OE/2600	Herbicide Clean-Up

4.6 Organic Analysis Department Standard Operating Procedures - Outline

SOP/OA/1000	Volatile Organic Compounds
SOP/OA/1200	Semi-Volatile Organic Compounds
SOP/OA/1300	Pesticides
SOP/OA/1400	Herbicides
SOP/OA/1500	PCBs

4.7 Quality Control Standard Operating Procedures – Outline

SOP/QC/1000	Thermometer Calibration & Inventory Management
SOP/QC/1005	Applying Thermometer Temperature Correction Factors
SOP/QC/1020	Fume Hood Inspection
SOP/QC/1500	Document Control & Revision Tracking
SOP/QC/2000	Corrective and Preventative Actions
SOP/QC/3500	QA/QC Data Review & Reporting

4.8 Sample Control Standard Operating Procedures - Outline

- Statement of Purpose
- Introduction
    - Sample Custodian - Duties
    - Sample Control Standard Operating Procedures
    - Sample Labels
    - Sample Seals
    - Analytical Services Request Form

Chain-of-Custody Procedures and Documentation
Delivery, Receipt, Inspection and Logging of Sample
Assignment of Analysis
Preservation
Holding Time
Internal Sample Request Form
Sample Disposal

- List of Figures and Tables
  - Figure 1. Sample Control Flowchart
  - Figure 2. Sample Label Example
  - Figure 3. Analytical Services Request Form
  - Figure 4. Chain-of-Custody Form
  - Figure 5. Sample Control Irregularity Stamps
  - Figure 6. Analytical Request Worksheet-Organics & Metals
  - Figure 7. Analytical Request Worksheet-Inorganics & Non-Metallics
  - Figure 8. Internal Sample Request Form
  - Figure 9. Sample Refrigerator Log
  - Figure 10. Reagent-Grade Water Conductivity Log
  - Figure 11. Analytical Balance Calibration Log
  - Table 1. Environmental Sampling Guide

4.9 Personnel Policy Manual - Outline

- Hiring Policies
  - Non-Discrimination Policy
  - Probationary Period
- Hours of Work
  - Hours of Work
  - Overtime
- Benefits
  - Paid Holidays
  - Vacation Eligibility
  - Health Insurance
  - Dental Insurance
  - Life Insurance
  - Long Term & Short Term Disability Insurance
  - Mileage Reimbursement
  - Personal/Sick Days
  - Funeral Leave
  - Military Leave
  - Leave of Absence due to Medical Disability
- Pay
  - Paydays
- Working Conditions
  - Health and Safety Regulations

Job Related Injuries
Dress code
Smoking
Conduct on Company Property
Drug Testing
Jury Duty

- Personnel Evaluation & Promotion
  - Probationary Period Evaluation
  - Merit Evaluation
  - Criteria for Employment Evaluation
  - Promotions
- Resignations and Terminations
  - Resignations
  - Terminations
- Employee Harassment
- Employee Open Door Grievance Procedures
- Amendment

4.10 Chemical Hygiene Plan - Outline

- Preface
- Introduction
- General Lab Safety
- Personal Hygiene
  hand washing, sniffing chemicals, mouth pipetting, smoking, eating, drinking, food, clothing, jewelry, cosmetics, shoes, hair
- Personal Protective Equipment
  eyes, gloves, lab coat, aprons, distance, respirators, estimation of risk
- Good Laboratory Practice
  horseplay, exits, radios, housekeeping, equipment, stools, standards, glassware, procedures, working alone, unattended operations, emergency procedures
- Protective Equipment in the Lab
  signs, labels, hoods, emergency lighting, other equipment, maintenance
- Electrical Safety
  introduction, grounding, electrical emergencies, inspections, fluids, extension cords
- Chemical Hazards
  introduction, knowledge, MSDS, types of hazardous chemical classifications, hazard identification system, Right-to-Know, "A Lot is Up To You", current MSDS sheets
- Safe Handling of Chemicals in the Workplace
  sample handling, hazard identification, labeling, handling toxic chemicals, corrosive materials,reactive/unstable chemicals, flammable liquids, pyrophoric chemicals, hypergolic mixtures, combustible liquids, carcinogens, reproductive toxins, compressed gases, chemical storage, chemical inventory
- Waste Disposal
  disposal of solvents, acids, corrosives, hazardous materials, sample consolidation, glass
- Chemical Emergency Response Plan

prevention, spills, containment, clean up, decontamination, reporting

- Evacuation Plan
- General Fire Safety
  fire extinguishers, classes of fires, other fire equipment, housekeeping, flammable liquids, clothing fires, fire exits, ignition sources, location of equipment, using an extinguisher, fire emergency plan
- Severe Weather Precautions
  tornado, flood, gas leak, power outage
- First Aid
  introduction, eye wash/showers, first aid kits, emergency procedures
- Emergency Phone Numbers
  emergency equipment, hospitals, trauma centers, poison, control center, CHEMTREC, hazardous spill control
- Record Keeping
  exposure record, medical record, training/education log, incident log, accident report form, inspections

5.0 LABORATORY PRACTICES

5.1 Project Scheduling and Acceptance

FIRSTECHNOLOGY evaluates all new projects and work to determine if adequate resources are available to fulfill the needs of the client. The Laboratory Director, along with the sales staff and QA/QC will evaluate the scope of work required. Any existing contracts should be periodically reviewed to ensure contract requirements are still being met. The following items are considered when undertaking new work or reviewing existing contracts:

- Is the proper equipment available to perform the analyses required? The equipment must be suitable for the test methods requested by the client and/or regulatory authorities to meet the scope of the project.
- Can the samples be processed in a timely manner to meet client deadlines and/or sample holding times when introduced into the regular laboratory workload?
- Is adequate, qualified staff available to perform the testing in a timely manner?
- Are there adequate laboratory supplies available to start the project on time? If not immediately available, can supplies be ordered and received on time?
- Can the laboratory adequately meet all project QC requirements? Are any changes required to the existing quality system, and does this compromise any existing QC requirements?
- If certifications are required for the new work, does the laboratory possess such certifications, and if so, do existing certifications fully cover all work required?
- Can the laboratory perform the specified analytical methods?
- Would the new project or work conflict in any way with existing projects or work?
- Will any subcontract work be necessary? Does the lab have an adequate subcontractor? Is the subcontracting acceptable to the client?

Records are maintained of the review and acceptance of new work, as well as periodic review of existing contracts.

5.1.1 Subcontract Work

Contracted laboratories that perform analyses for FIRSTECHNOLOGY are required to follow quality guidelines in line with the requirements of the FIRSTECHNOLOGY quality program. Subcontractors used must be accredited according to the requirements of the work subcontracted. FIRSTECHNOLOGY is responsible for the work of subcontract labs, except when client requirements specify a particular subcontractor.

All laboratory reports are flagged to identify subcontractors, either by name or accreditation number. FIRSTECHNOLOGY maintains a list of all subcontractors, and quality documentation on each subcontractor.

5.2 Sample Management during Collection/ Shipment

FIRSTECHNOLOGY tries to provide as much information to field personnel as possible. In an attempt to provide this information regarding sampling techniques, bottles, labels, and chain of custody documentation, the laboratory has prepared a Guide to Sampling and Sample Management. Information pertaining to all aspects of sampling, from documentation to transport to the laboratory, is contained in this document.

5.3 Handling Samples in the Laboratory

Labels
All samples are labeled upon receipt with a sample number. This identification does not cover any information on the labels concerning field information or collection information. This identification uniquely identifies every sample in the laboratory. Bottles containing different aliquots of the same sample are identified with a unique bottle ID number. The combination sample number and bottle ID is used in laboratory notebooks and logs to provide traceability to what bottle and sample was used for a specific analysis.

Container Type, Quantity, and Preservative
Each analyst is responsible to verify the container type and preservative required by the method being performed. The analyst must use the glass container if the method requires a glass container to be used in the collection of the sample. Furthermore, the correct preservative must be used in the preparation of the sample. If the bottle is not preserved in the field, it will be preserved upon receipt by the lab based on the analytical tests requested. The analyst must verify the correct preservation of the sample before using the aliquot.

Any alteration that the analyst makes to a sample must be done on an aliquot of the sample. Preservative cannot be directly added to a bottle that has multiple analytical parameters requested. For example, if a single sample bottle is marked for pH, BOD, and TSS and the BOD requires adjustment of the pH as described in the method, the entire bottle must not have a pH adjustment unless the pH and the TSS have already been performed. An aliquot of the sample must be taken and correctly labeled with the sample identification and the alteration that was made, in addition to the analysts initials, date, and time. A new bottle ID is assigned to the new aliquot and recorded on the sample Bottle ID Login form. In the remarks column of the Bottle ID form, the analyst must indicate which bottle ID the aliquot was taken from, and date and initial the change.

Any login irregularities should be noted during login. Date and initials must be provided to document any irregularities discovered when logging new samples.

Sample Storage
Samples are stored in designated sample storage refrigerators while awaiting analysis. Samples that do not require refrigeration may be kept in Sample Receiving at room temperature. There is a separate refrigerator to store VOC samples away from other samples to prevent contamination. Refrigerators used for sample storage are subject to daily temperature checks to verify they are operating at the correct temperatures. Samples are kept a minimum of 30 days from the issuance of a laboratory report. Samples may be kept longer at a customer's request. Analysts must return samples to sample storage following analysis.

Representative Samples
Each analyst is responsible for obtaining a representative aliquot of the sample for analysis. It is recommended that a representative sample for solids should be obtained by cone and quarter technique. This technique involves removing the entire sample from the jar, mixing it thoroughly and then mounding the sample and slicing the pile in equal quarters. This task is performed until the appropriate size aliquot is obtained for analysis. To obtain representative samples for liquids, samples should be thoroughly mixed. Liquid samples with particulates should be mixed in such a manner to sample the particulates equally in each aliquot.

Meeting Holding Time Requirements

Holding time is based on the date of collection rather than the verified time of sample receipt, and date of digestion / distillation. It is a technical evaluation rather than a contractual requirement. Technical requirements for holding times have only been established for water matrices. When holding times are exceeded the reviewer of the data must use professional judgment to determine the reliability of the data and the effects of additional storage on the sample results.

FIRSTECHNOLOGY makes every effort to comply with holding time requirements; however we cannot guarantee compliance to these regulations if samples are not received at the laboratory within 24 hours of collection.

Security

FIRSTECHNOLOGY employs a chain of custody procedure for sample transfers. Sample transfers are documented with a signature, date, and time. This documentation occurs when a customer transfers custody to our laboratory as well as other custody transfers that occur before samples become the custody of the sample custodian within the laboratory. In addition to chain of custody transfer documentation, sample access and handling is controlled. Only authorized personnel are permitted in sample holding areas. Samples in storage are kept in refrigeration units where their integrity can be maintained.

Optionally, at the request of a customer, FIRSTECHNOLOGY can provide documentation of chain of custody once samples are in the possession of the laboratory. Samples can be stored in a lock box within sample refrigerators, and an internal custody transfer record is maintained between analysts and the sample custodian. Analysts requiring samples for analysis under these circumstances are expected to complete an internal request for samples. The transfer of samples is documented on these forms.

5.4 Glassware Washing Procedures

Scrupulously clean glassware is essential to all laboratory procedures. The excuse of 'contaminated glassware' to explain a failed analysis is unacceptable and cannot be used. Glassware cleanliness must be assured regardless of the other problems that arise during subsequent laboratory functions. The following are specific steps that laboratory personnel must perform when washing glassware for general purpose.

1) Place dirty glassware on the appropriate bench in the glassware washing room. If you cannot wash the glassware immediately, the containers should be filled with tap water to limit adhesion of contaminants to the vessel walls. Do not store dirty items on the clearly marked clean glassware benches.
2) Scrub each vessel thoroughly with a brush, Alconox detergent and warm tap water.
3) Rinse the glassware with enough tap water that the rinse water runs off cleanly. The walls of the glassware must drain evenly with no channeling of the water on the walls. If there is channeling, the item is not clean and a repeat of step 2) is necessary.
4) After the tap water rinse, rinse the item at least three times with reagent-grade water so as to thoroughly displace the tap water.
5) Rack the item for efficient draining and air drying. As soon as the items are dry they should be stored in the appropriate cupboards. If the glassware remains in the room too long it may be contaminated by airborne dust.

After the glassware has been prepared as above, special procedures such as acid rinsing can then be utilized, however, these special procedures are not substitutes for the general preparation.

5.4.1 Special Cleaning Requirements

The following cleaning techniques are quite useful for special parameters.

Organic Parameters
Where the presence of trace concentrations of organic matter may present a problem (e.g., BOD, COD, pesticides), a chromic acid cleaning solution is used. The glassware is immersed in a chromic acid solution or it is filled and allowed to stand for 15-20 minutes. After soaking, the glassware is rinsed thoroughly with warm tap water and then reagent-grade water. Glassware to be used in the BOD test must be free of chromate. Glassware to be used in pesticide analyses is also rinsed with pesticide grade acetone.

Trace Metal Parameters
Before first use, glassware is soaked in 50% nitric acid for two days then in hydrochloric acid for two days. The rinses consist of warm tap water and reagent-grade water. This glassware is stored separately from all other glassware after cleaning. Subsequent daily usage require only rinsing with 50% nitric acid.

Nutrient Parameters
Glassware for use in the analysis of nitrogen or phosphorus parameters is only washed with 1:1 hydrochloric acid. Detergents are never used. The glassware is rinsed with tap water and reagent-grade water and stored separately.

Pipets are washed in the following manner. They are loaded tips up into a pipet washer. Next, the washer is filled with hot detergent water where the pipets soak for a minimum of 30 minutes. Finally, they are put through a series of rinsings with tap water and reagent-grade water respectively.

5.5 Purchase, Purity, Storage, and Preparation of Purchased Reagents, Standards, and Laboratory Supplies

Analytical Reagent Grade Water
Reagent-grade water is used in the laboratory for preparation of reagent solutions, dilutions, and final rinsing of glassware. FIRSTECHNOLOGY has a Barnstead Ropure LP (reverse osmosis) with NANOpure polishing system to provide the highest purity analytical reagent-grade water. It is monitored daily for conductivity. Any conductivity greater than 2 umhos/cm at 25 °C is unacceptable and the ion exchange system is replaced immediately.

Requirements of Reagents
Reagents purchased must be analytical reagent grade or ACS grade or spectral grade and must meet or exceed the requirements of the method. Reagents ordered should be in minimum quantities so that they will be consumed within manufacturer's expiration date or within six years for solids and within a year for liquid reagents (except for pH buffers), whichever is first. pH buffers should be stored no longer than six months after opening. Any reagents that have deteriorated are not used for analysis. Deterioration includes abnormal caking, moisture absorption, a change in color, or a change in composition or physical state.

The date of receipt, date of first opening and the expiration date are duly recorded on each container.

Standard Solutions and Titrants
All solutions that are prepared in house are to have the following information of the label.

1. Name of Solution
2. Unique Lab Identification Number
3. Concentration of the Solution
4. Date solution was prepared
5. Date solution expires
6. Initials of the person who prepped the solution

Finally the solution should be stored in an acceptable container. Volumetric flasks do not qualify as acceptable containers.

Unless specified by the method, standard solutions or titrant will not be held longer than one year.

All prepared standards must be fully documented in the appropriate Reagents & Standards logbook. The standard must be given a unique Lab identification number and the following information recorded:

1. Date of receipt of purchased material used to prepare standard
2. Date prepared
3. Name and final concentration of standard
4. Quantity of standard prepared
5. Solvent used for dissolution or dilution
6. Reagent (and its concentration) used to prepare standard, including the manufacturer and lot #
7. Expiration date
8. Preparer's initials

All anhydrous reagent chemicals required for preparation of standard calibration solutions and titrant are dried in an oven at 105°C for at least two hours and overnight if possible. Should a different drying temperature be necessary, this is specified for the particular chemical. After cooling to room temperature in an efficient desiccator, the reagent is promptly weighed for dissolution. For hydrated salts, suggested drying procedures are used instead of oven-drying.

Preparation of standard solutions or titrant from a primary standard is formulated in volumetric flasks when exact concentrations are required. Solutions are mixed thoroughly and completely when dilutions are made. A common source of error in standard formulations in volumetric flasks is the failure to attain complete and homogeneous mixing. Where exact concentration is not required, the solutions are formulated with graduated cylinders. Standard solutions or titrant not prepared from a primary standard are stabilized against the primary standard at the frequency specified by the method, or every six months if no frequency is specified. Unless specified by the method, standard solutions or expiration date and storage conditions are listed on the label. At any time that significant degradation is detected the solutions are discarded and re-formulated.

Purchased Standard Solutions and Titrant

Purchased prepared standard solutions must have a certification of the contents and the concentration, a statement that the preparation was in accordance with approved methodology, and an expiration date. All purchased calibration standards must be traceable to NIST standards.

All routine quality control practices use independent sources of reagents. The new supplies of standard solutions or titrant are checked to show the equivalent responses as the existing stocks. Second source standards are also used to check the primary supplier's formulations and QC procedures.

The laboratory purchases prepared standard solutions and titrants when the preparation of these solutions are too time consuming. Manufacturers or suppliers of the preparations must provide:

1. certification of contents and concentration
2. statement that preparation was in accordance with an approved methodology
3. expiration date

Bench or Shelf Reagents
Bench reagents should contain the similar information on the bottle as that of standards. These reagents are also recorded in a bound logbook stating the similar information as that of standards. In addition the drying times and temperatures, the weights or volumes used and as appropriate the calculation to adjust solution to nominal concentrations, and final standardization titers and calculations. All reagents must be traceable to the material in which they were made from.

Bench reagents are those that do not act as a standard or titrant in an analysis. These solutions are prepared by the laboratory or purchased from independent suppliers. Documentation for these solutions follows the procedures above.

Record of Reagents, Titrants, and Solutions
In a bound logbook, the following are recorded (example in APPENDIX D):

1. Name and concentration of solution or titrant
2. Lab Identification number for solution
3. Manufacturer and lot number of reagent-grade chemicals used
4. Quantity of solution prepared
5. Appropriate expiration dates
6. preparer's initials

and as appropriate:

1. drying times and temperatures
2. gross, tare and net weight
3. volumes used
4. citation of preparation of primary
5. calculations to adjust solution to nominal concentration
6. final standardization titers and calculation

5.5.1 Laboratory Suppliers and Supplies

The FIRSTECHNOLOGY Laboratory maintains a current list of approved vendors. These numerous vendors are required to fulfill the laboratory needs in a cost effective and timely manner.

The supplies procured by the laboratory are required to meet the quality criteria in terms of purity, certificate of analysis, expiration date, and any other general and/or technical information for handling and disposal of the material.

As for ordering of lab supplies, the laboratory personnel submit a purchase requisition form to the lab manager for approval. After approval, the requisition is forwarded to the person(s) in charge of ordering.

5.6 Obtaining and Reporting of Detection Limits

The method detection limit is defined as the minimum concentration of a substance that can be identified, measured and reported with a 99% confidence that the analyte concentration is greater than zero, and determined from analysis of a sample in a given matrix containing analyte.

Procedure for Determining the Method Detection Limit (MDL)

1 Make an estimate of the detection limit using one of the following:
   - 1.1 The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5.
   - 1.2 The concentration value that corresponds to three times the standard deviation of replicate instrument measurements of a reference standard.
   - 1.3 The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations.
   - 1.4 The concentration value that corresponds to known instrumental limitations.

2 Using reagent blank water prepare a laboratory control sample at a concentration between 1 and 5 times the estimated detection limit.

3 Take a minimum of seven aliquots of the sample and process each through the entire analytical method.

4 Calculate the standard deviation of the replicate measurements.

5 Then calculate the MDL as follows:

MDL = Student T Value X standard deviation

Student T Values	
Number of Replicates	**Student T Value at the 99% Confidence Level**
7	3.143
8	2.998
9	2.896
10	2.821

MDLs are determined at least annually and more frequently if the method and specific SOP requires it.

Methods with stated MDLs are verified annually to make sure that the lab can meet the required level of detection.

The analyst is required to keep a record of most current MDL study in addition of submitting a copy of the study to the QA Officer. The copy provided to the QA Officer will be kept for archive purposes.

Results determined to be less than the Detection Limit are reported as BDL (Below Detection Limit). All detection limits are reported on every parameter for analysis.

Raising Reporting Limits (RL)
Reporting limits should be increased if the analyst dilutes the sample because of matrix interferences or if there was a smaller than normal sample volume used in the analysis. This is most important when the sample is subsequently found to be BDL. Reporting limits should also be increased if the initial sample result exceeds the linear dynamic range of the instrument, requiring dilution.

Detection Limits for Solids
Results for solid sample should be reported in mg/Kg and the detection limit should also reflect the solid matrix of the sample as it was received. To obtain the solid detection limit the base liquid detection limit should be multiplied by the ratio of the final volume to the weight of the sample used:

$$\text{Base liquid DL (mg/L)} \times \left(\frac{\text{Final Volume (mL)}}{\text{weight of sample (g)}} \right)$$

5.7 Miscellaneous Laboratory Practices

Broken Glassware Procedures
Broken glassware is not to be used under any circumstance. This situation is a safety hazard but an also can lead to improper analysis as a result of trapped residuals in the cracks of glassware.

If you break glassware, you should be report the breakage immediately to the immediate supervisor and also complete the paperwork needed to replace or repair the break.

Purchasing Procedures
All requests for analytical reagents, supplies, and equipment should be made in writing on a requisition form to the lab manager.

Housekeeping
General laboratory cleanliness is the responsibility of the analyst. You are responsible for cleaning up after yourself. This should include but not be limited to: throwing away towels, Kimwipes, and papers, returning samples after completion of analysis, cleaning and proper storage of glassware, cleaning benchtops, keeping glassware washing room clean, and removing of excess waste jars from storage areas.

6.0 LIST OF MAJOR ANALYTICAL EQUIPMENT

6.1 Inorganics/Wet Chemistry

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
Westco Easy-Dist System	Westco Scientific	Easy-Dist	1144	12/2005
SETA Multiflash Flash Point Tester	Stanhope-SETA	34100-2	1002110	2006
SEAL AQ2+ Automated Discrete	SEAL Analytical, Inc.	AQ2+	090535	1/4/2006
IR Spectrometer	Perkin-Elmer	1600	136542	1989
TOC Analyzer	Dohrmann	3300	N9C5563T	1989
TOX Analyzer	MCI	TOX-10	43C31419	1989
Calorimeter	Parr	Parr Adiabatic Calorimeter	P1490073	1989
Spectrophotometer UV/VIS	Milton Roy	Model 21D	3151155003	1989
Wet Chem Ion Analyzer	Orion	EA920	TW26A	1989
Turbidimeter	Monitek	Model 21	L-3575	1989
DO Meter	Orion	850	28241	1997
Pensky-Martens Flashpoint Tester	Precision Scientific, Inc	74537	10AX-4	1989
Muffle Furnace	Thermolyne	62700	1075980485736	1997
Sonicator	Branson	Sonifier 250	BH70128	1997
Conductivity Meter	YSI	Model 31A	C9015656	1989
Wet Chem Analytical Balance	Denver Instrument	M-310	N0081085	1998

6.2 Laboratory

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
Water Purification System	Barnsted	ROPure-NANOpure	55000552	1989

6.3 Metals Laboratory

Instrument Group HP ICP-MS

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
HP ICP-MS	Hewlett-Packard	4500	3622J 00559	2003

Instrument Group Varian Graphite Furnace AA

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
Varian Graphite Furnace Autosampler	Varian	GFAA Autosampler	9091069	1989
Varian Graphite Furnace	Varian	Zeeman Furnace	QTA962-9091105	1989
Varian AA-400	Varian	SpectrAA 400	AA400Z-9091036	1989

Instrument Group Varian ICP

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
ICP Autosampler	Varian	SPS-5	94101466	1999
Varian ICP	Varian	Liberty AX II	EL99043684	1999

Instrument Group Varian Mercury Flame AA

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
Varian AA-20 Plus Flame AA	Varian	SpectrAA 20 Plus	1091446	1989
Varian Flame AA Autosampler	Varian	PSC-56	1091348	1989
Varian Mercury Cold Vapor Unit	Varian	VGA-76	1091399	1989

6.4 Organics Analysis

Instrument Group HP GC with Dual ECD #1

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
HP Autosampler	Hewlett-Packard	7673A	3120A26667,	1989
HP Gas Chromatograph (GC)	Hewlett-Packard	5890 II	2413A06027	1989
HP Dual ECD	Hewlett-Packard		M2166,F3636	1989

Instrument Group HP GC with Dual ECD #2

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
HP Gas Chromatograph (GC)	Hewlett-Packard	5890 II Plus	3310A49322	2005
HP Dual ECD	Hewlett-Packard		K0749, M1697	2005
HP Autosampler	Hewlett-Packard	6890	3529A43288,	2005

Instrument Group HP GC with Dual FID

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
HP Autosampler	Hewlett-Packard	7673A	2933A11485,	1989
HP Gas Chromatograph (GC)	Hewlett-Packard	5890 II	2938A25578	1989
HP Dual FID	Hewlett-Packard			1989

Instrument Group HP HPLC System

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
HPLC Scanning Fluorescence Detectors	Waters	470	470-001595	2000
HP HPLC	Hewlett-Packard	1050	3127G02402	1998

Instrument Group Semi-Volatiles HP GC/MS System

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
HP Autosampler	Hewlett-Packard	7673A	2932A14207	1989
HP Mass Selective Detector (MSD)	Hewlett-Packard	5972	3524A02705	1999
HP Gas Chromatograph (GC)	Hewlett-Packard	5890 II Plus	3336A60140	1999

Instrument Group VOC GC/MS System #1: HP GC/MS

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
HP Mass Selective Detector (MS)	Hewlett-Packard	5972	3329800705	2005
HP Gas Chromatograph (GC)	Hewlett-Packard	5890 II	32335845762	2005
Varian Archon Autosampler	Varian	Archon 4552	13464	2000
Purge-and-Trap System	Tekmar	LSC 2000	92112012	1989

Instrument Group VOC GC/MS System #2: Varian Saturn 2000R GC/MS

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
Varian Saturn Gas Chromatograph (GC)	Varian	3800	07156	2001
Varian Saturn Mass Selective Detector	Varian	Ion Trap 2000	04758	2001
Varian Archon Autosampler	Varian	Archon		2001
Sample Concentrator	Tekmar/Dohrmann	3100	01017006	2001

6.5 Organics Extractions

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
Top Loading Balance-Organic Prep	Ohaus	Precision Plus	4719	1999

6.6 TCLP Extractions

InstrumentName	Manufacturer Name	Model	SerialNo	Date Rec.
Top Loading Balance-TCLP	Ohaus	Navigator	D188008479	1999
pH Meter-TCLP	Corning	320	C11171	1999

7.0 INSTRUMENT CALIBRATION

7.1 Major Equipment

Every piece of major equipment has a manufacturer's operating manual available.

Every piece of equipment that requires calibration is monitored to verify the frequency of the calibration, date of last calibration, when the calibration is due, and what the calibration source is. These guidelines are established according to procedures specified by the manufacturer's manuals and methods manual. These maintenance requirements and calibrations are logged for each major instrument.

In addition to this, the name and location of every piece of equipment is kept in an equipment list inventory. Preventative Maintenance schedules are followed according to the manufacturer's guidelines or whenever a problem exists. Any defective equipment is removed from service until an authorized representative of the manufacturer corrects and inspects the piece of equipment. Once equipment is operational, a calibration and verification of the calibration must be performed before any analysis can be performed.

The calibration record maintained with the instrument indicates the date of calibration and date of expiration in addition to the initials of the analyst performing calibration.

Instrument Calibration
Instruments are calibrated according to method requirements and manufacturer documentation. Before and after each set of samples, the calibration is checked with a calibration verification check sample. Additional QC is run based on method requirements. Calibrations that can be re-used over time are not used for more than three months.

If any problems or irregularities are discovered during the above procedure, immediate corrective action is taken. The procedure and equipment is tested with certified standard reference materials to isolate the problem. If the problem is in the procedure or due to experimental error, the problem is corrected, the sample re-extracted if applicable and the sample set is rerun with full quality control checks.

7.2 Support Equipment

All support equipment to performing analysis is maintained and calibrated on a regular schedule according to the specific guidelines listed below.

Balances

Analytical balances are available for all analytical work which requires sensitivity to 0.1 mg. They are serviced by a qualified service organization annually and will display a service sticker with the date of service. Balances will be checked on a weekly basis with class S weights in two ranges. The bound, record logbook of this QC check contains the date of check, the target and actual readings and the analyst's initials.

pH Meters

The pH meter is available with sensitivity to ± 0.05 and is calibrated with each use of the calendar day. It is calibrated with two pH buffers within the working range and then verified with a buffer at the end of the operating range which must agree within ± 0.05 pH units. Records of the calibration and slope are kept in the pH logbook.

Thermometers

The thermometers within the company are checked on an annual basis with an NIST thermometer. The NIST thermometer is checked annually at the ice point. The date of inspection, the reading, adjustment to be made to the correction factor, the new correction factor and the analyst's initials in tabular format is kept in a bound logbook kept with the QC officer. Then each thermometer is checked against the NIST thermometer. This record of the thermometer number, calibration temperature, correction factors and analyst's signature is kept in a bound logbook with the QC officer.

Reagent Grade Water

Daily the conductivity of the Reagent grade water is checked and recorded in a bound logbook next to the instrument. Reagent water should be less than 2.0 umhos/cm at 25°C.

Oven/Furnaces

The oven/furnace temperature is checked at the beginning and end of each cycle of use. The temperature logbook is kept with each oven and the date, target temperature, time and temperature of the start and the time and temperature of the end is recorded as well as the purpose of its use and the initials of the analyst.

Refrigerators/Incubators

Refrigerators and incubators containing samples and/or standards are verified twice daily (at least four hours apart) for temperature conditions. Records of the temperature are kept in a bound logbook of this QC check and contain the date of the check, the temperature, and the analyst's initials in addition to the identity of the Refrigerator or incubator that was checked.

Volumetric Glassware

Class A volumetric glassware is used by the laboratory.

Spectrophotometer

The spectrometer is calibrated every six months. The record of the calibration of the Spectrophotometer is kept in the maintenance logbook next to the instrument. This bound logbook contains the record of calibration and maintenance that takes place on the instrument.

Conductivity meter and cell

The conductivity cell constant will be determined weekly using a 0.01M potassium chloride solution. The record of this cell constant is kept in the reagent grade water logbook and contains the date of this QC check, the resistance readings, the average resistance, as well as the water temperature, calculations and initials of the analyst.

The conductivity meter's calibration is also verified with each calendar day of use with an NIST traceable standard. This record is kept in the conductivity meter logbook.

Dissolved Oxygen Meter

The dissolved oxygen meter is calibrated with each calendar day of use. This is done by filling three BOD bottles with aerated distilled water. The dissolved oxygen concentration is determined in two of the bottles and the third is used to calibrate the meter at the average D.O. found. This record is kept in the bound BOD logbook with the date, the D.O. values, the average D.O. and the analysts initials.

Turbidimeters

The turbidimeter is checked with each calendar day of use with formazin standard in the range of interest. The date, target values, observed values, and analyst's initial are kept in the turbidity logbook.

Background Lead Testing

On a quarterly basis the lab will perform wipe sample analysis of the lab work surfaces for lead. The results of this analysis will be documented with the QA Officer.

7.3 Other Laboratory Equipment

Magnetic Stirrer

Magnetic stirrers are available for all analytical procedures which require mixing. Stirrers are variable speed and are used with Teflon coated stir bars.

Hot plate

Hot plates are available for all analytical procedures which require heating. All heaters contain temperature control settings.

Centrifuge

A centrifuge is available with a revolution rate up to 3000 rpm.

Color Standards

Color standards are available to verify wavelengths on photometers and cover a range from 200 to 800 nm.

Drying Oven

A drying oven is available for convection heating and drying with a temperature range from room temperature to 300°C.

Water Bath

Several water baths are available which provide heating to 100°C and are controllable to within 5°C.

8.0 QUALITY CONTROL ANALYSIS

8.1 Method Blank

The laboratory method blank consists of reagent-grade water or solvent or carrier gas which is processed and analyzed as if it were a sample using the same glassware, reagents and known additions. A laboratory method blank is run daily or with each batch of twenty samples and needs to be labeled with a day or batch identifier. (i.e., MB-4/9/96). In addition, if multiple prep procedures are performed in one day, a method blank needs to be performed with each preparation type and each group of twenty samples.

The method blank is calculated in the same manner as a sample and a tabulation of blank results for each analyte with the data are run and the acceptance criterion is reported.

The assessment of method blank analysis is to determine the existence and magnitude of contamination problems. Method Blanks are not subtracted from the samples. The only exception is in the analysis of colorimetric tests where the method requires a **color** blank subtraction based on each individual sample.

8.2 Field Blanks/Trip Blanks

Trip and field blanks are required by certain GC methods. Trip blanks are reagent grade water placed in a sample container at the laboratory and remains unopened through the entire trip. The trip blank determines whether the samples have been contaminated by the shipping procedures used to transport the samples to the site and back to the laboratory. A field blank represents reagent grade water that is placed in a sample container at the site or is filled in the laboratory and travels to the site, opened and re-sealed at the sampling site. They are then shipped to the laboratory with the environmental samples taken. A field blank should be included with sample collection at each sampling site. Field blanks are to be analyzed when samples from a specific sampling site are positive for the analyte of interest. If reportable levels of the analytes of interest are demonstrated to have contaminated the field blank, re-sampling is required.

8.3 Laboratory Control Sample (LCS)

An LCS sample is analyzed once per preparation batch. A preparation batch is 20 or less samples.

LCS samples are prepared according to method-specified requirements.

An LCS sample control chart is constructed and control limits are updated annually. The control limits are calculated from the standard deviation, or the following general limits are used:

Warning Limit = mean ± 2 Standard deviations
Control Limit = mean ± 3 Standard deviations

Control charts are used detect shifts and trends in method performance. If control charts show serious shifts in performance, maintenance or troubleshooting should be initiated.

8.4 Initial and Continuing Calibration Verification

Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid. This calibration verification may be required to be from a second source, meaning that the standards utilized to verify the calibration are from another source and prepared independently of the calibration. Consult specific test methods as to the requirements of the source of the ICV/CCV.

Continuing calibration verifications must be run throughout the analysis run at no less than every ten samples. The precision must be within 10% or the sample batch back to the last good CCV must be rerun. This is considered the same as the ICV just analyzed throughout the run rather than initially.

Initial and continuing calibration must fall between 90 - 110% unless otherwise specified by the relevant analytical method. All initial calibrations are verified with a second source standard.

8.5 Matrix Spike/Matrix Spike Duplicate

A matrix spike represents an aliquot of a sample that has a known concentration of the compound being analyzed added to the aliquot in an attempt to determine the matrix affects on the recovery of the desired compound being analyzed.

Spiked environmental samples are analyzed at a frequency of one spiked sample for every 10 samples or batch. Spiked recovery for an environmental sample is determined by dividing the sample into two to three aliquots depending on regulations. The first aliquot is analyzed as usual. The second and third aliquots are spiked with a known concentration of the analytes of interest.

The spike volume should be less than 5% of the aliquot volume. This means if a sample aliquot is 100 ml, less than 2 ml of spiking solution should be added to spike the sample.

For long lists of analytes, use the following guidelines for choosing a representative number of analytes to spike. Representative analytes will be rotated periodically. All target compounds should be used in a spike mixture over a two year period.

For 1-10 analytes, spike all analytes
For 11-20 target analytes, spike at least 10 or 80%, whichever is greater
For greater than 20 target analytes, spike at least 16 analytes.

Spike analysis should not be performed on field or trip blanks. In addition, when the concentration of the analyte in the sample is greater than 1000 ppm, no spike of the analyte is necessary.

Limits for acceptable matrix spike results are determined from statistical calculation of in-house samples.

As a general rule until control limits are established spikes must be within 75% - 125% for Inorganics and Metals. Recommended guidelines are provided in the organic methods. However, spike recovery limits do not apply when the sample concentration exceeds the spike concentration by a factor of four or more. If a spike recovery is outside recovery limits, and the sample concentration does not exceed the spike concentration by a factor of four or more, a post-digestion/post-preparation spike should be prepared and reanalyzed.

8.6 Surrogate Standard

A surrogate standard is a compound or a mixture of compounds which, when spiked into an environmental sample, behaves in the same manner as the analyte or analytes of interest. When analytes are determined by GC and GC/MS procedures, the surrogate standards are used to determine chemical recoveries from environmental samples. When surrogate standards are applicable to a method, the sample is spiked with a surrogate standard unless a specific exception is made in the method itself. Percent recovery for each surrogate standard is calculated, tabulated and control limits established.

A surrogate standard chart is constructed for each surrogate for every twenty five samples analyzed. The control limits are calculated from the standard deviation of the percent recoveries and charts constructed in the same manner as matrix spike data.

8.7 Field Duplicates

A duplicate field sample is an analysis performed at the same time on equal quantities taken from the same sampling point. Duplicate analysis data provides information about the data variation due to sampling. Duplicate analysis is required of 10% of all positive samples for a given analyte.

8.8 Duplicate Data

A duplicate analysis is an analysis performed on equal quantities from the same aliquot of sample. Duplicate analysis data provides information about the data variation due to sampling not only in the field but how an analyst obtains a representative sample. RPDs must be charted for all parameters.

As a general guideline until RPD are calculated for all parameters, RPD should not exceed 20% for liquids and 35% for solids or soils if the results of the sample are greater than five times the detection limit. If the results do not exceed the five times the detection limit the range of the duplicate should be $\pm$ DL for liquids or $\pm$ twice the detection limit for solids.

A sample labeled as a field or trip blank cannot be used for duplicate sample analysis.

A range of duplicates control chart is constructed each analyte for every twenty five samples analyzed. In the chart of duplicate data, the range (difference) is determined. The acceptable limit is calculated as

$$\text{Upper Control Limit} = 3.27\ R$$

In addition to the range, the Relative Percent Difference is calculated as well.

8.9 Standard Curves

In general, standard curves must be composed of a minimum of a reagent blank and three standards covering the concentration range of the samples being prepared. One of the standards should be half of the maximum contaminant level and one standard should be equal to the MCL. In addition the correlation coefficient must be greater than 0.995. Any day that the curve is used, it must be verified by a lab method blank and one standard near the MCL. This daily check must be within 10% of the original curve. The metals department must make a curve on every day of analysis which must be made as stated above.

8.10 Interference Check Standards

Interference check standards are used by the metals department to determine the effects of potential interfering elements. A standard is analyzed containing only potential interferences, and then a standard is analyzed containing potential interferences with analytes of interest. Comparison of the two standards can determine if interference correction is necessary.

8.11 Contamination Control Samples

For required contamination control documentation, specified lab areas will be checked for surface contamination. This applies to Lead and PCB wipe testing. On a quarterly basis, designated lab surfaces are checked using sample wipes. The following areas are checked:

Lead:

Counter-top around the analytical balance
Counter-top of the metals prep area
Hood area used for metals prep
Sample log-in location

PCBs:

Counter-top around sample balance used for weighing
Counter-top used for sample prep
Hood area used for sample prep
Sample log-in location

8.12 Corrective and Preventative Actions

Corrective actions are taken when laboratory data and/or procedures used depart from standard operating procedures as covered in this document and specific method documentation. Preventative actions are taken to eliminate the cause of a potential problem. Preventative actions are taken to prevent an occurrence of a problem in the first place. See SOP/QC/2000 for further details on corrective and preventative actions.

Certain corrective actions can be found within specific method SOPs. Analysts are required to be able to assess their QC data, to realize when QC results deviate from criteria established by the FIRSTECHNOLOGY Quality System, and when to alert management of potential problems

The final authority on corrective actions is with the QA Manager. If an analyst is unable to resolve a QC problem using normal, documented procedures, a Testing Irregularity Report (Form QC-2010) is filled out and reported to the QA Manager along with laboratory data. The following items must be addressed:

1. The person(s) responsible for initiating and taking responsibility for corrective action
2. Evaluation of the significance of the nonconforming work
3. Corrective actions taken immediately; evaluation of the acceptability of data.
4. Record of notification of the client if necessary
5. Person(s) responsible for the resumption of work.

The QA Manger will review the Irregularity Report to determine what actions need to be taken. By reviewing the totality of performed QC, the QA Manager may determine that the QC is acceptable and may be reported without further action. Review will determine if the problem necessitates halting analysis, if data is able to be reported, and if the customer was notified of the issue. If results were reported prior discovery of nonconforming work, amendments and qualifications to the original report may need to be made. All of these decisions and justifications are documented on Form QC-2010.

For general irregularities that are not specific to testing, such as equipment out of spec (Incubators, Refrigerators, etc.) Form QC-2005 is used to document the irregularity. This form is used to document weather sample integrity was affected, if clients were notified, and if results can be reported. A follow-up attachment includes details on preventing future problems, and what is being done to correct the problem.

The QA Manager may also consult with the Laboratory Director on QC issues if the situation warrants it.

If QC data is still out of control, re-analysis may be necessary to achieve the required quality of results. If for any reason (samples beyond holding time, insufficient sample volume for re-analysis, instrument problems preventing re-analysis) samples cannot be re-analyzed, the customer should be notified and informed of the deviations from normal QC.

The customer may decide on resampling or accepting data as-is. If data from an out-of-control situation is reported, the results should be flagged with appropriate comments and data qualifiers. See Section 11.3.9 for data qualifiers that may be used to flag out-of-control data.

9.0 METHOD GUIDELINE QC REQUIREMENTS

9.1 Metals Analysis

9.1.1 Drinking Water

GFAA

- Initial calibration must be done each day and each time instrument is set up. Must consist of a blank and at least three standards, one of which must be at the detection limit to establish analytical curve. R-value 0.995 or greater.
- Analyze a Reference sample every 10 samples or quarterly if fewer than 10 samples received in the quarter.
- An Initial Calibration Verification (ICV) is run immediately following calibration. The recovery should be between 95 – 105%.
- A Reporting Limit Check (RLC) standard should be analyzed at the beginning of the analytical run. The recovery should be between 70 – 130%. The standard concentration is the reporting limit of the analyte of interest.
- MS/MSD performed with a frequency of one in ten or fewer samples. Recovery for positive samples above the RL is 80 – 120%, and recovery for samples below the RL is 70 – 130%.
- After every ten samples, analyze a CCB and CCV. The CCV should be between 90 – 110%. A CCB and CCV should be run at the end of the run also.
- Method blank each batch (not to exceed 20 samples)
- Duplicate performed each batch (not to exceed 10 samples)

ICP/ICP-MS

- Initial calibration must be done each day and each time instrument is set up. Must consist of at least one standard and a blank to establish analytical curve.
- An Initial Calibration Verification (ICV) is run immediately following calibration. The recovery should be between 95 – 105%.
- A Reporting Limit Check (RLC) standard should be analyzed at the beginning of the analytical run. The recovery should be between 70 – 130%. The standard concentration is the reporting limit of the analyte of interest.
- Analyze a reference sample with every 10 samples or quarterly if less than 10 samples in analytical batch.
- Method blank each batch (not to exceed 20 samples)
- If digestion takes place due to turbidity readings, a LCS must be prepared and analyzed.
- Duplicate performed each batch (not to exceed 10 samples)
- A matrix spike must be analyzed each batch and every ten samples and recover within 85-115% for positive samples and 70-130% for samples below the RL.
- After every ten samples, analyze a CCB and CCV. The CCV should be between 90 – 110%. A CCB and CCV should be run at the end of the run also.

Mercury

- Initial calibration must be done each day and each time instrument is set up. Must consist of a blank and at least four standards, one of which must be at the detection limit to establish analytical curve. R-value 0.995 or greater.
- An Initial Calibration Verification (ICV) is run immediately following calibration. The recovery should be between 95 – 105%.
- A Reporting Limit Check (RLC) standard should be analyzed at the beginning of the analytical run. The recovery should be between 70 – 130%. The standard concentration is the reporting limit of the analyte of interest.
- A reference sample with each batch (not to exceed 10 samples)

- Method blank with each batch (not to exceed 20 samples)
- LCS every 10 samples and for each digestion
- Duplicate performed each batch (not to exceed 10 samples)
- A check standard or calibration standard must be at the MCL value.
- An MS/MSD must be analyzed each batch and every ten samples and recover within 85-115% for positive samples and 70-130% for samples below the RL.
- After every ten samples, analyze a CCB and CCV. The CCV should be between 90 – 110%. A CCB and CCV should be run at the end of the run also.

9.1.2 Liquid Analysis of Metals

GFAA

- Initial calibration must be done each day and each time instrument is set up. Must consist of a blank and at least three standards, one of which must be at the detection limit to establish analytical curve. R-value 0.995 or greater.
- Initial Calibration Verification (ICV) – 90 - 110%
- Reference sample run with at the beginning of the run. 90 – 110% recovery limits.
- MS/MSD analyzed with each batch and every ten samples and recover
- Duplicate in each batch (not to exceed 10 samples)
- Method blank each batch (not to exceed 20 samples)

ICP

- Initial calibration must be done each day and each time instrument is set up. Must consist of at least one standard and a blank to establish analytical curve
- Second Source reference standard every batch (not to exceed 10 samples)
- Spike in each batch (not to exceed 10 samples) with recoveries between 75 - 125%
- Method blank each batch (not to exceed 20 samples)
- Duplicate performed every 10 samples of the same matrix type of more frequently if less than ten samples in batch
- Calibration verification must be analyzed every ten samples or at the beginning and end of each batch if fewer than ten samples.
- LCS run with each digestion sample type and spiked with every sample being analyzed. Recovery with 85 - 115%

Mercury

- Initial calibration must be done each day and each time instrument is set up. Must consist of a blank and at least four standards, one of which must be at the detection limit to establish analytical curve.
- Analyze a reference sample with every 10 samples or fewer if less than 10 samples in analytical batch.
- Method blank every 20 samples or monthly if fewer than 20 samples received in the month
- Duplicate performed each batch (not to exceed 10 samples)
- A matrix spike each batch (not to exceed 10 samples) and recover within the following determined ranges:

  - TCLP extracts 70 - 130%
  - Solids 87 - 113%
  - Liquids 78 - 122%

- Calibration verification must be analyzed every ten samples or at the beginning and end of each batch of ten or fewer samples.

9.1.3 Solid Waste Analysis for Metals

- Reference Sample every ten samples or monthly
- Spike on at least 10% of all samples
- Duplicates must be done on at least 10% of all samples and preferably every sample
- All other QC as designated by instrument

9.1.4 TCLP Analysis for Metals

- Spikes must recover at least at 50%. If the contaminant measured is within 20% of the regulatory level, the spike must be done by methods of standard additions.
- All other QC as designated by instrument.

9.2 Inorganic Analysis

9.2.1 Drinking Water Inorganics

- Analyze reference sample every 10 samples or monthly if less than 10 samples received in the month.
- Analyze a lab control sample with each batch with recoveries between 90-110%
- Method blank every twenty samples or monthly
- Duplicate performed every ten samples
- Matrix spike performed when applicable on 10% of the samples with recoveries between 90 - 110%.

9.2.2 Solid/Liquid Analysis of Inorganics

- **BOD**
- Method blank each batch
- Duplicate each batch (not to exceed 10 samples)
- (same dilution on the same sample)
- Reference sample every batch
- Seed Control daily

- **COD and TOC**
- Method blank every batch of samples
- Reference sample each batch (not to exceed 10 samples)
- Spike every 10 samples or once per batch (75 - 125%)
- Duplicates each batch (not to exceed 10 samples)
- (20% RPD)

- **TS, TSS, TDS**
- Method blank every batch of samples
- Duplicates each batch or every 10 samples (20% RPD)

- Standard verification on a monthly basis

- **Oil and Grease**
- Method blank every batch of samples
- Standard each batch or every 10 samples
- Duplicate each batch or every 10 samples
- Reference sample every quarter
- Spike every 10 samples or once per batch (75 - 125%)

- **Cyanide, Phenol**
- Method blank every batch of samples
- Standard sample every batch or 10 samples
- Spike each batch or every 10 samples (75 -125%)
- Duplicate each batch or every 10 samples
- Curve must be made up of a blank and at least three standards
- A mid-range standard must be distilled. (LCS 85 -115%)

- **Surfactants**
- High and Low Standard every 10 samples or monthly
- Spiked sample each batch or every 10 samples
- Duplicate each batch or every 10 samples

- **Alkalinity, Acidity, Fluoride**
- Standard each batch and every 10 samples or monthly
- Verification of Acid Normality
- Duplicate each batch or every 10 samples
- Method blank each batch or every batch of samples

- **Ammonia, Nitrate, Nitrite, Phosphorus all forms**
- Standard each batch and every tenth sample or monthly
- Spike each batch or every 10 samples
- Duplicates each batch or every 10 samples
- Method blank every batch of samples

- **Nitrate/Nitrite**
- Cadmium column efficiency must be determined each time a column is used and should be within required guidelines.
- Ohio EPA Drinking Water: 90 - 110 %

- **All other inorganic parameters for liquids and solids**
- Reference sample every 10 sample or monthly
- Spiked sample each batch or every 10 samples
- Duplicate each batch or every 10 samples
- Method Blank in each batch
- Distilled or prepared standard in each batch

9.3 Organic Analysis

9.3.1 Drinking Water THMs

- Field blank to be analyzed with positive results
- Laboratory blank to be analyzed with each batch or daily
- Reference sample to be analyzed every 10 samples or monthly
- Duplicates on 10% of samples.

9.3.2 Drinking Water Volatiles

- Field blank to be analyzed with any positive results
- Lab blank to be analyzed daily or with each batch and must not exceed the laboratories method detection limit
- Reference sample to be analyzed every 10 samples or monthly
- Duplicates on 10% of samples
- Low level Reference sample
- Calibration must be verified at the beginning of each day on which analysis is performed with a reagent blank and one standard in the expected concentration range of the samples analyzed that day. All checks must be within control limits or the system recalibrated.

9.3.3 GC/MS Water/Wastewater Volatiles

- Initial calibration at the beginning of each analytical batch at concentrations of 10, 20, 50, 100, and 200 □g/L and as necessary if CCV criteria is not met. Note: Separate CCV are analyzed for water and for low level solid samples due to the fact that water samples are performed on an unheated purge and that soil samples are heated.
- Relative Response factors and System monitoring compounds must be greater than or equal to 0.05.
- The % RSD from the initial calibration must be less than or equal to 30.0% for all compounds.
- Lab blank daily or with each batch
- Reference sample every 20 samples or monthly
- Spiked sample each batch or every 20 samples
- Duplicates each batch or every 10 samples
- Surrogate on all samples

9.3.4 GC Water/Wastewater Volatiles

- Lab blank daily or with each batch
- Reference sample every 10 samples or monthly
- Spiked sample each batch or every 10 samples
- Duplicates each batch or every 10 samples
- Surrogate on all samples

9.3.5 GC/MS Water/Wastewater Extractables (BNA)

- Lab blank daily or with each batch
- Reference sample every 20 samples or monthly
- Spiked sample each batch or every 20 samples

- Surrogate on all samples
- Duplicates each batch or every 20 samples

9.3.6 Drinking Water Pesticides/Herbicides

- Laboratory Blank daily or with each batch
- Reference Sample with every 10th sample of monthly if less than ten samples received.
- Duplicates on 10% of samples.
- Method blank every twenty samples or monthly

9.3.7 GC Water/Wastewater Pesticides

- Lab blank daily or with each batch
- Reference sample every 10 samples or monthly
- Spike sample every 10 samples or monthly

10.0 STATISTICAL QUALITY CONTROL (SQC) & DOCUMENTATION

There are two basic indicators of measurement of quality, namely precision and accuracy.

Precision refers to the reproducibility of a method when it is repeated on a homogeneous sample under controlled conditions, regardless of whether or not the observed values are widely displaced from the true value. This is the result of the systematic or constant errors present. However, when evaluating results from validated methods, the final estimate of precision will usually rely on the assumption that all practicable steps have been taken to control error. The remaining fluctuations are considered random and determine the experimental precision.

Accuracy refers to the agreement between the amount of a component measured by the test method and the amount actually present. In other words, it is a measure of how near the result is to the true value. The best method to verify accuracy is to add a known amount of the substance being measured to an aliquot of the actual sample and determine the percent recovery of that known addition.

Relative error expresses the difference between the measured and actual amounts as a percentage of theoretical amounts. Thus, the relative error represents the measure of accuracy of a method.

10.1 Standard Curves

Standard curves are to be prepared as specified in the methods. All standard curves are dated and labeled with the method, analyte, standard concentrations and instrument responses. The instrument data acquisition system calibrates by drawing a best fit, straight line and calculating the correlation coefficient by the least-squares method. An acceptable correlation coefficient is 0.995 or greater. Instrument response for samples is less than the highest standard and the lowest standard is at the detection limit. The calibration curve is verified with an initial calibration verification (ICV) prepared from the primary standard source.

If a method does not provide guidance in the preparation of a standard curve, the following guidelines are followed. For manual colorimetric methods, a blank and five standards that lie on the linear portion of the curve is derived. New curves are constructed whenever the response of the previous curves no longer fall within method regulations, SQC-derived limits or when new standards are formulated. At each use, the curve is checked with a low initial calibration verification (LICV) and a high initial calibration verification (HICV). The HICV selected is to be higher than the expected sample concentrations. These standards are prepared from the primary standard source.

10.2 Matrix Spike (Known Addition) Percentage Recovery Calculations

A qualitative estimate of the presence or absence of interfering substances in a particular determination may be made by a means of a matrix spike recovery procedure. The percent recovery, %R is calculated as follows:

$$\%R = 100 \times \frac{(S_t - B)}{T}$$

where:

S_t = The total measured concentration of total analyte in the spiked sample aliquot.
B = The background concentration of the unspiked sample aliquot.
T = The target value i.e. known concentration of analyte spiked into the sample aliquot.

The analysis date and percent recoveries of the spiked samples are recorded. When a minimum of twenty matrix spikes have been tabulated, the mean percent recovery is computed. The standard deviation and warning limits are computed and recorded.

A tabulation of surrogate percentage recoveries is also maintained. The tabulation includes the analysis date, the percent recovery and the control limits for %R for each surrogate.

The percent recovery, %R is calculated according to:

$$\%R = 100 \times \frac{M}{T}$$

where:

M = The measured value
T = The target value (i.e. the known value of surrogate spiked into the sample).

Quality control is used to monitor the precision and accuracy of laboratory measurements and the sampling process. Internal quality control is established by routinely analyzing control samples as laboratory blanks, field blanks, reference standards, spiked environmental samples, surrogate standard and duplicate analysis. External quality control is performed by participating in the USEPA Performance Evaluation programs as well as programs administered by customers and other agencies.

10.3 Statistical Quality Control (SQC) Charts

An SQC chart is generated for particular analytes as required by the regulatory agencies. The applicability of SQC chart techniques is based on the assumption that the laboratory data approximate a normal distribution. Measured values are accumulated as part of a day to day operation. At least twenty data points are used to compute acceptance limits. A new tabulation with freshly calculated limits are computed every six months.

Using the mean, the standard deviation (Sd) is calculated using the formula below:

$$Sd = \sqrt{\frac{(X - \bar{X})^2}{n-1}}$$

where:

X = The measured value of an individual reference standard

$\bar{X}$ = The mean of the measured values
n = The number of data points

The upper and lower control limits (UCL and LCL) are +3 and -3 standard deviations from the mean, respectively, and the upper and lower warning limit (UWL and LWL) at +2 and -2 standard deviations.

A control chart is constructed and the measured values of the reference standard samples are then plotted on the chart (see APPENDIX E).

$\bar{X} + 3\ Sd$ ———————— UCL, 99%

$\bar{X} + 2\ Sd$ ———————— UWL, 95%

$\bar{X}$ ———————— Central Line, mean

$\bar{X} - 2\ Sd$ ———————— LWL, 95%

$\bar{X} - 3\ Sd$ ———————— LCL, 99%

Should one result fall outside the control limits (the 99% confidence limit), two successive results fall outside the warning limits (the 95% confidence limit) or 5 successive results fall either above or below the central line, the analysis is out-of-control and immediate action is taken to determine the cause of the outlying result. If the cause cannot be identified and corrected, data generated on the same day as the outlying result are regarded as unreliable and the analysis repeated after corrective action has been taken and the procedure is back in control.

10.4 Duplicate Analysis and Matrix Spike Determinations

A duplicate analysis is required when a sample yields a positive result. Matrix spikes and matrix spike duplicates are required on at least a one in twenty schedule but are treated the same as other duplicate analyses.

The difference (range) between duplicate analyses is determined as follows:

$$R = | X_1 - X_2 |$$

where:

R = the difference (or range)
X_1 = the first measurement
X_2 = the second measurement

Range control charts are generated by computing the mean of the ranges recovered and graphing limits equal to 0.845*mean, 2.456*mean and 3.27*mean. These factors relate to the 50%, 95% and 99% confidence limits respectively. (Actual examples can be found in APPENDIX F). Should one result be above the 99% line, two successive results fall above the 95% line or five successive results fall above the 50% line, the analysis is out-of-control and again the problem is remedied before further analyses are run.

$\bar{R} * 3.27$ ———————— CL, 99%

$\bar{R} * 2.456$ ———————— WL, 95%

$\bar{R} * 0.845$ ———————— 50%

$\bar{R}$ ———————— Mean

10.5 Precision and Reproducibility

Precision is defined as the measurement of agreement of a set of replicate results among themselves without assumption of any prior information as to the true result. Precision is measured by determining the agreement between sample duplicates expressed in terms of relative percent difference (RPD).

RPD is calculated by the equation:

$$\text{RPD} = \left(\frac{(S - D)}{\left(\frac{(S + D)}{2} \right)} \right) \times 100$$

where:

S = sample concentration
D = duplicate concentration

Charting of QC Analyses

A means chart is constructed for laboratory control standards. In this chart the true value or average is considered the base line and then acceptable limits both above and below the average are calculated using the the ±2*SD (warning limits) and ±3*SD (control limits)

Charts are maintained by the quality control officer on all data submitted for Quality Control review.

11.0 DATA PROCESSING AND REPORTING

11.1 Data Management, Documentation and Archiving

The laboratory keeps records of submitted samples and completed analyses in a manner that provides for complete sample traceability and data retrievability. Information such as the source of the sample, matrix type, preservation, initial pH, initial temperature, sample site, and the manner in which the sample was shipped or delivered to the laboratory are recorded on the chain-of-custody and kept with the permanent record. This information is also found in the sample receipt logbook. The persons responsible for the sampling are also documented.

All manually quantified results are recorded in bound logbooks with unique ID numbers. The pages of the logbooks are lined in a grid pattern with provision for such information as sample identification, extraction date, detection limit and analyst as well as other information. Also included are references to standards and reagents used to provide traceability to materials used for calibration and analysis. All entries are permanent. No erasures are permitted. Any change or correction is initialed and dated by the person making the change. These manually quantified results are reported to quality assurance personnel on specifically designed report forms (examples in APPENDIX C).

When the data are produced by the use of an automated system, the data are displayed by means of a computer printout which lists the FIRSTECHNOLOGY sample number, the actual date and time of analysis. The analyst supplies his/her signature, sample preparation conditions and the applicable operational parameters of the instrument or procedure. Also included are references to standards and reagents used to provide traceability to materials used for calibration and analysis.

As stated previously, FIRSTECHNOLOGY employs a computerized LIMS to log, track and report sample progress. This facilitates the storage of all sample identification information, analysis results and quality control data. All data generated by the laboratory including Chain-of-custody forms, instrument printouts, bench sheets, logbooks and final reports are filed and maintained. In addition, all data stored on computer media are retained as part of the permanent record.

11.2 Documentation

The lab keeps hard copy and computer records of submitted samples and completed analyses in a manner that provides for data retrievability on the sample preservation, the traceability of the sample source, the procedures, and the persons responsible for the sampling and analysis.

Permanent logbooks are kept which document actions within the laboratory. These logbooks contain grid lined bound pages which are sequentially numbered. These pages make provisions for sample identification, date, matrix, sample size, dilution, instrument readings, calculations, detection limit, spike or standard concentration and recoveries, and analysts initials. All entries must be made in permanent ink in sequential order and must be complete. No erasures, black outs, or correction fluid are permitted in any logbook. Any changes or corrections must have a single line drawn through the error and the change written above the line. Any change must be initialed and dated by the person making the change. Any unused pages or empty sections are crossed out with a single diagonal line through the unused portion. Quantified results are entered into the LIMS system by the analyst. This data entry is subject to review by the Quality Manager pending final discharge of results.

Sample receipt information such as the source of the sample, matrix type, preservation, initial pH, initial temperature, sample site, and the manner of shipment or delivery are recorded on the chain of custody and also in the permanent sample receipt logbook. Most of this information is also logged into the LIMS system. Currently, the lab uses a customized computerized LIMS system (Lablite) to log, track and report the progress of samples through the lab. This facilitates the storage of all sample identification information, analysis results and quality control data. All data generated by the laboratory including chain of custody forms, instrument printouts, bench sheets, logbooks and final reports are filed and maintained for a ten (10) year period. In addition all data stored on computer media are retained as part of the permanent record. Records are retained within the LIMS system. The LIMS system is periodically cleared of older data to maintain speed and performance when accessing the LIMS system. Older data are stored in separate databases, and may be loaded into Lablite at any time it is required. Typically data older than two years is archived.

Manually quantified results are recorded in bound logbooks. Information for the days entries must contain the procedure being utilized, the IDs of the samples analyzed, the date and time, extraction date, detection limit, and analyst's initials. In addition, any direct readings off an instrument must be recorded which will show any weights, volumes, or dilutions used in the analysis, and the concentrations of spikes added. A sample calculation for each batch must be shown. Finally all quality control samples analyzed and the calculations for appropriate recovery must be documented. This includes the calibration verification and the source of this verification. Any irregularities or changes in the SOP must be noted on irregularity forms.

Data produced by the use of an automated system, is displayed by means of a computer printout which lists the sample number, the date and time of analysis, the analysts name, sample preparation conditions and the applicable operational parameters of the instrument or procedure. A logbook with additional information concerning the weights, volumes, or dilutions used in the analysis, the concentration of spike added and the quality control samples analyzed in the batch accompanies the automated system.

11.2.1 Archiving/Back-up

All paperwork generated by the laboratory including chain of custody forms, instrument printouts, notebooks and final reports are filed and maintained according to regulatory requirements. All records are retained to allow for a complete reconstruction of all data from login to analysis to final reports. The following storage requirements are followed:

- For Ohio EPA Drinking Water: General chemistry and laboratory records are retained for a period of 10 years. (OAC 3745-81-33)
- For Ohio EPA Drinking Water: Records pertaining to the Lead and Copper Rule are retained for a period of 12 years. (OAC 3745-81-90)
- NELAC Requirements: Per NELAC Quality Systems regulations, records are retained for a minimum of five years unless designated for a longer period by another authority or regulation. (NELAC Quality Systems Rev 16, 5.4.12.2.4b; NELAC Accreditation Process Rev 15, 4.3.3)
- Discharge, hazardous waste, and all other records will follow NELAC requirements unless otherwise specified by another authority or at a specific client request.
- Records involved in litigation must be maintained until written authorization is received that all litigation is closed and records can be disposed of.
- All computerized records are retained as part of the permanent record.

11.2.1.1 Log Book Tracking

For tracking laboratory notebooks and logbooks, the following items are assigned to logbooks:

- A unique ID is assigned to each book when issued, using the following format:
  - LOG-<yyyymmdd##>
    - The ID uses the date of notebook creation, plus a two digit number if more than one notebook is created on the same date.
- Logbook Title/Description
- Date of Issuance by QA/QC
- Book type, such as Data or Maintenance
- Date of return to QA/QC
- Archive Location: ID storage box used

The QA/QC department uses a Microsoft Access database to track logbook locations.

11.2.2 Security

FIRSTECHNOLOGY employs a computerized LIMS database to log, track and report sample progress. There are levels of security which protect the database from unauthorized users. The LIMS is accessible only with proper login procedure and password. Back-up of all files is completed nightly. In addition, FIRSTECHNOLOGY has a battery back-up system which provides for two hours of computer use in which time the system could be completely backed-up if needed. The system memory is contained on disks and tapes which are currently archived in the system operator's office. Future plans are being made to store these tapes off the premises in a secured fire-proof vault.

11.2.3 System Administration and Maintenance

FIRSTECHNOLOGY employs consultants for network maintenance of software as well as maintenance of the hardware. Printers are serviced annually and work stations are monitored on a quarterly basis for repairs as needed.
11.3 Tracking of Samples and Analysis through the Laboratory

11.3.1 Login

From the time the samples are relinquished to the Sample Custodian, logbooks are utilized at all points in the sample trail. Immediately upon arrival, the sample is logged in and the following information is recorded in a bound logbook by the sample custodian.

- Customer Name, Customer Sample Identification
- Assigned FIRSTECHNOLOGY Sample ID
- Date and time received
- No. of containers and container type
- Preservation in each bottle
- pH and temperature upon receipt
- Sample matrix
- Storage refrigerator
- Special sample disposal request
- Login person's initials

11.3.2 Data entry / Verification

Once samples have been logged into the laboratory manually, the chain of custody and analytical request information is manually entered into the LabLite LIMS system. This information is then reviewed by the Report Coordinator for accuracy and completeness.

11.3.3 To-Do Lists

The LabLite LIMS system generates To-Do Lists which allow analysts to review work to be done. Custom To-Do Lists are used to display work required for specific analysts and departments. The Report Coordinator can generate new lists as necessary.

11.3.4 Analysis/ Raw Data

Analysis is performed on samples as specified by the To-Do Lists from LabLite. Information is collected in logbooks maintained by analytical personnel. Entries into logbooks are made according to the specifications made in section 5.2 entitled Documentation. Full analytical instrument printouts may be substituted for a logbook provided sufficient information is available on the printout.

11.3.5 Initial Data Entry

Once a requested analysis is performed on a sample batch, the results are reviewed and entered into LabLite, where they will be reviewed for precision and accuracy. The raw data is then filed with the sample chain of custody form.

11.3.6 QC Checks

On completion of a laboratory sample, the QA Officer will review results and the report for completeness, accuracy and precision, as well as looking for data transcription errors and verifying calculations. All sample results are completely traceable and the train in which the results are generated can be totally reconstructed including all hard copy of chromatograms and other instrument printouts. If all data is acceptable, the report is approved for printing. If there is a problem, the report is rejected and corrective action is started.

11.3.8 Printing and Reporting

The report is printed and signed by the QA Officer, who then will fax, phone, or email preliminary results to the customer. The final report is then reviewed by the lab manager. The lab manager then reviews and signs the report and sends the final copy to be sent out by mail.

Final reports list the analyte results, detection limits, method numbers, hazardous waste codes, surrogate recoveries, and other QC parameters.

When a report is amended, a comment should be placed on the report that it is amended, and refer to the date of the original report.

11.3.9 Data Qualifiers

When the analysis or samples have some type of irregularity that does not meet methodology requirements, it is important to place data qualifiers on the results when they are submitter for report generation. Whether these codes are used or a comment is written is not important, however it is extremely important that the people generating the report are notified of these irregularities. For more complex situations, an attached detailed case narrative detailing is used to inform clients of irregularities.

J = estimated value due to a tentatively identified compound or the data indicates the presence of a compound that meets the identification criteria but the result is less than the specified detection limit. It can also be used to note that surrogate recovery limits have been exceeded, the value failed to meet established QC criteria, or the sample matrix interfered with the ability to make accurate determinations.

B = Analyte was detected in the blank as well as the sample.
L = The actual value is greater than the value given.
Q = The sample exceeded accepted holding time requirements.
Y = The analysis was from an unpreserved or improperly preserved sample. Data may not be accurate.
R = Quality control indicates that the data is unuseable. Re-sampling and or re-analysis is necessary for verification

11.3.10 Release of Analytical Report

Under no circumstances should individual analysts be in contact with customers, salespeople, or consultants about sample results.

We have requested that all members of the sales and consulting staff refrain from asking an individual analyst directly about an analytical assignment for their respective customers.

The release of analysis is made by either the QC officer and/or the Lab Manager. Analytical reports are signed by both individuals.

11.3.11 Confidentiality of Results

All results obtained from samples from any client are strictly confidential and will not be released to anyone except the client. To assure conformance with this policy, all requests for analytical information are to be addressed to the laboratory director or a designated representative.

11.3.12 Final Written Reports

Final written reports list parameter results as well as detection limits, method numbers, hazardous waste codes, surrogate recoveries and other QC parameters (APPENDIX G). FIRSTECHNOLOGY will also custom design report forms where possible to satisfy particular client reporting requirements.

11.3.12.1 Faxing Reports

When faxing results to clients, a fax cover letter accompanies all reports. The following cover letter message is intended to preserve confidentiality of results:

This is confidential message, intended soley for the person to whom it is addressed. If you receive this message in error, please forward it to the correct person, or mail it back to us. Thank you.

In addition, the following statement appears on the cover letter covering the status of the faxed reports:

The following report(s) contain preliminary results issued by FIRSTECHNOLOGY to expedite procedures to clients. FIRSTECHNOLOGY assumes that all sampling methods and data, upon which these results are based, are supplied accurately by the client. These results may not have been reviewed by the Laboratory Director. Final Reports of Analysis with Chain-of-Custody will follow these preliminary results by mail. The original signed Report of Analysis is considered the official report.

11.3.12.2 E-mailing Reports

FIRSTECHNOLOGY can provide e-mail copies of reports if requested by a client. Reports are only e-mailed after being reviewed by the QA/QC Officer. Reports are provided in Adobe PDF file format. The following signature comment accompanies all e-mailed reports to preserve the confidentiality of results:

This email and any attachments are confidential, intended solely for the person(s) to whom they are addressed. If you receive this message in error, please destroy the email and any attachments or copies. Please do not retain, distribute, disclose or use any of this information.

In addition, the following statement also appears covering the status of the email reports:

Any attached reports contain preliminary results issued by FIRSTECHNOLOGY to expedite procedures to clients. FIRSTECHNOLOGY assumes that all sampling methods and data, upon which these results are based, are supplied accurately by the client. These results may not have been reviewed by the Laboratory Director. Final Reports of Analysis with Chain-of-Custody will follow these preliminary results by mail. The final signed Report of Analysis sent by regular mail is considered the official report.

11.3.13 Billing

Billing is done through the computer generated billing system of *QuickBooks*. This billing is verified using the analytical request on the chain of custody.

11.3.14 Filing

Once billing and analysis is complete, the chain of custody is filled alphabetically by the customer name and stored on site for that calendar year. (January - December) Once that year is complete, chain of custody, raw data and preliminary results are boxed and archived. Some data may also be stored on electronic media, and is retained with the rest of the data. Section 11.2.1 details requirements for archiving and disposal of records.

11.3.15 Sample, Chemical, and Solvent Disposal

Once analysis is complete, samples, chemicals, and solvents are disposed of according to the disposal protocol as listed in the chemical hygiene plan.

12.0 ADDITIONAL QUALITY ASSURANCE ITEMS

12.1 Certifications

Drinking water by Ohio EPA
Wastewater and solid waste by New York DOH ELAP program

12.2 External Audits

12.2.1 New York DOH On Site Inspection

Site visits for recertification purposes are performed every two years. This is an announced site visit scheduled by the New York DOH. During this inspection various aspects of the laboratory's operation are reviewed:

- Verification of lab director's employment.
- Verification of lab location
- Evaluation of facilities in relation to workload
- Review of labs methods manual
- Review of Labs QC/QC plan
- Review of QA/QC records
- General record-keeping
- Evaluation of instrumentation and reagents for individual analytes and procedures
- Any other factors which might affect the quality of lab results.

12.2.2 Ohio EPA On Site Inspections and Requirements

All personnel performing analysis of drinking water must be certified by Ohio EPA. Each analyst is required to perform at least 10% of the analytical workload for drinking water. Each analytical test must be performed once every three months if the sample load does not require analysis more frequently.

Scheduled on site surveys are normally scheduled every three years. These full surveys will cover all aspects of analyses for which certification is sought. In addition, random unannounced surveys will be conducted during normal working hours 8:00 am - 5:00 pm when laboratory facilities and records must be available. During an on site survey a variety of tests may be scheduled to be performed during the evaluation. The evaluator will examine the procedures and equipment used for which the lab has requested certification. The evaluator will also review the records and standard operating procedures and insure that the QA plan is in effect.

12.3 Annual Internal QC Audit

An internal audit will be conducted annually by the staff of FIRSTECHNOLOGY or an appointed designee thereof. This audit will review all aspects of the laboratory. Assessment by outside agencies may also be used for internal audit information. The annual internal audit will be completed during the 1st Quarter of the new year following the completion of all outstanding work for the previous year. See Sec 12.9 for items to be reported to management.

12.3.1 Logbook Inventory

The following lists the inventory of all logbooks kept by the laboratory. Random inspections take place on all logbooks at least every two weeks. Logbooks must be signed and dated by the person inspecting them.

12.3.2 Bi-Weekly Inspections

- Sample Custodian (Temperature & Maintenance)
  - Sample Storage Refrigerators (Units 1 & 2)
  - Oven (Unit 1) (Note: Unit 2 used only for glassware)

- Wet Chemistry and TCLP Extractions
  - Analytical Balances (Units 1, 2 & 3), Calibration & Maintenance
  - NanoPure Water System Conductivity (includes conductance meter cell-constant determination)
  - Lachat Auto-analyzer, Maintenance
  - Spectronic 21D
  - Reagents and Standard Storage Refrigerator (Unit 2)
  - Other
    - Reagent and Standard Preparation (front Wet Chem laboratory)
    - Reagent and Standard Preparation (cyanides, phenols, sulfides, bromides)

- Metals
  - Calibration & Maintenance (ICP, ICP/MS, GFAA, Flame AA)
  - Mercury Water Bath Temperature (Unit 4)
  - ICP Emission Check
  - Reagents and Standard Storage Refrigerator (Unit 1)
  - Reagent and Standard Preparation

- Organics - GC/MS VOCs
  - Calibration & Maintenance
  - Reagent and Standard Preparation
  - Sample Storage Refrigerator (Unit 3)
  - Reagents and Standard Storage Refrigerator (Unit 5)

- Organics - GC/MS BNAs
  - Calibration & Maintenance
  - Reagent & Standard Preparation
  - Reagents and Standard Storage Refrigerator (Unit 4)

- Organics - GC
  - Calibration & Maintenance (one book for all units)
  - Reagent and Standard Preparation (one book each for GC/ECD, GC/FID, GC/Hall/PID)

Reagents and Standard Storage Refrigerator (Unit 6)

- Organic Extractions
  Reagent and Standard Preparation
  Reagents and Standard Storage Refrigerator (Unit 3)

12.3.3 Random Inspections

- Other Logbooks
  Sample Login, Wet Chem Analyses, TCLP Extraction, Metals Digestion, Mercury Digestion, ICP Analysis, ICP-MS Analysis, GFAA Analysis, Flame Analysis, GC/MS VOC Analysis, BNA Extraction, GC/MS BNA Analysis, Pest/PCB Extraction, Herbicide Extraction, Pest/PCB Analysis, Herbicide Analysis

12.4 Proficiency Evaluations

The laboratory currently participates in proficiency evaluations for Ohio Drinking Water Studies and New York DOH Waste Water and Solid Waste performance evaluations. In addition, DMR-QA samples are received by certain treatment plant operators for parameters analyzed for their testing programs. Proficiency samples are accepted semi-annually by New York and by APG for the Ohio EPA Drinking Water Program. Drinking water proficiency results are passed on annually to the Pennsylvania and Kentucky programs.

The proficiency samples are received by the Quality Assurance officer, and then logged into the laboratory as a sample. To maintain certification as a certified laboratory, the lab must satisfactorily analyze PE samples and maintain an overall proficiency rating as determined by each individual certification program.

Proficiency samples are handled in a similar manner as normal client samples. The State of New York DOH requires each analyst sign a statement to this extent.

Following receipt of proficiency results, the QA/QC Officer evaluates all results and determines what corrective actions are needed. Form QC-2015 is used to distribute and document corrective actions for proficiency testing.

12.5 Internal Blind Samples

FIRSTECHNOLOGY analyzes at least two proficiency studies in a year for NPDES discharge, drinking water, and solid waste. If FIRSTECHNOLOGY is not required to analyze at least two studies in a year for an area, the laboratory will voluntarily purchase and analyze a set of blind samples to meet the laboratory minimum requirements of two studies per year.

Once results are complete, the data is interpreted by the QA Officer and the lab manager for completeness, accuracy, and precision. Results of all blind sample results are available with the QA Officer.

In addition, further studies may be undertaken to correct any deficiencies found during internal or external audits, or failures on required proficiency samples.

12.6 Quarterly Data Audits

On a quarterly basis laboratory notebooks and randomly selected data sets are checked for accuracy and complete performance. They are checked for documentation, correct data entry, calculations, calibration, data transcription,

report format, and chain of custody documentation. Laboratory notebooks are checked for accuracy and logged as inspected for that quarter. Notebooks will be examined for possible violations of the FIRSTECHNOLOGY Data Integrity policy. Also, a set of analytical samples (6-10 sets) will be fully audited to check for compliance with Data Integrity Procedures and the requirements of this document.

Quarterly audits will be performed in the first month following the end of the quarter.

In addition systematic review of a data set to identify outliers, suspect values, systematic errors, and transcription errors is performed on a daily basis with each analytical report.

12.7 External Inspections

Inspections and audits are performed frequently throughout the year by our customers and potential clients. Any client recommendations or deficiencies found during an inspection of the lab is reviewed for relevance to the laboratory operation. If a change in policy or procedure is deemed necessary, the appropriate corrective action and documentation is implemented.

12.9 Report of Quality Assurance to Lab Management

On a quarterly basis, the Quality Assurance Officer will submit, in writing, a summary report to the Managing Director regarding any and all Quality Control problems encountered during the month, the corrective actions taken, and the results of any audit deficiencies.

In addition, any assessment of measurement of quality indicators, results of performance audits, results of system audits and any significant QA problems and recommended solutions.

The following items should be assessed during the annual management reviews:

- The suitability of policies and procedures: are all procedures being followed adhering to required certifications, and maintaining the integrity of the quality system.
- Reports from managerial and supervisory personnel
- Outcome of recent internal audits
- Corrective and preventative actions reviewed: these actions should be documented and filed according to procedure; these records are reviewed to assess their effectiveness.
- Assessments by external bodies: includes certification agencies as well as customer audits
- Results of interlaboratory comparisons or proficiency tests: corrective action may be implemented after assessment
- Changes in volume or type of work
- Client feedback: review filed customer inquiry forms
- Client complaints: review filed customer inquiry forms
- Other relevant factors, such as quality control activities, resources, staff and training.

12.10 Drinking Water Contingency Plan

This plan is to be implemented for guidance, on Ohio EPA drinking water samples, when the completion of the required analysis will not be completed in a timely manner due to **any** circumstance.

1. If applicable, have the analyst complete an irregularity report explaining the nature of the delay and the anticipated remediation date.

2. Call and inform the client of the current situation and the expected return to normalcy date. Allow the client the option of subcontracting the work to another certified facility or hold the sample(s) until the corrective action has been completed.
3. If the work is to be subcontracted, contact the potential subcontract lab(s) for pricing and turnaround time. Inform the client of the options, i.e., cost, turnaround time, OEPA reporting fees, etc.
4. If the work is to be subcontracted, send the sample(s), with an analytical request and chain-of-custody, *via* UPS (overnight if required due to holding time issues) to the subcontract lab. Also, if the subcontract lab is to report results to OEPA, provide the subcontract lab with all appropriate reporting information (e.g., PWS ID, STU ID, client name, address, contact person, phone number, sample collection date/time, etc.)

12.11 Documentation of Customer Complaints

FIRSTECHNOLOGY maintains Customer Inquiry Forms to document customer concerns/complaints when they occur. These forms indicate the date the inquiry occurred, the name of the customer, the contact person, samples involved, a description of the nature of the inquiry, and a section describing what steps were taken to resolve the inquiry.

These Customer Inquiry forms are filed and are used during annual management reviews and QC audits.

13.0 SAFETY AND THE CHEMICAL HYGIENE PLAN

Physical conditions in the laboratory comply with the applicable Occupational Safety and Health Act (OSHA) requirements. The staff of FIRSTECHNOLOGY, Inc is instructed and encouraged to follow the guidelines for safety as established in the standard operating procedures as well as the Chemical Hygiene Plan.

Each employee on an annual basis is required to review the Chemical Hygiene Plan and renew his or her commitment to safety in the laboratory.

13.1 Periodic Testing/Monitoring – Leak Testing

For safety, electron capture detectors on GC instruments are tested annually for radioactivity leakage. This is a requirement of instrument maintenance.

14.0 SUMMARY

FIRSTECHNOLOGY, Inc., being a full-service environmental laboratory, uses stringent measures to assure that the results supplied to the customer are accurate, precise and complete. The QA program is designed to not only document that procedures are working as developed but to identify and remedy out-of-control conditions immediately upon detection. In this way, the end user can have high confidence that all information has been successfully validated and verified.

15.0 REFERENCES

APHA-AWWA-WPCF, 1989. Standard Methods for the Examination of Water and Wastewater, 17th ed.

American Society of Testing and Materials, 1988. Standard Guide for Good Laboratory Practices in Laboratories Engaging in Sampling and Analysis of Water. D3856-88, ASTM, Phila. 10 pp.

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DeRoos, Fred, M. Bicking and W. Nosek Jr., 1990. Some Precautions in the Analysis of Organic Materials. *American Env. Lab.* 10:16-24.

Dux, James, 1986. Handbook of Quality Assurance for the Analytical Chemistry Laboratory. Van Rostrand Reinhold, New York. 123 pp.

Keith, Lawrence A., 1990. Environmental Sampling: A Summary. *Environ. Sci. Technol.*, 24:610-617.

Taylor, John, 1987. Quality Assurance of Chemical Measurements. Lewis Publishers, Chelsea, MI., 328 pp.

United States Environmental Protection Agency (USEPA), 1983. Methods for the Chemical Analysis of Water and Wastes, 600/4/79/020.

USEPA, 1985. Methods for the Organic Chemical Analysis of Municipal and Industrial Wastewater. 40 CFR 136 Appendix C.

USEPA, 1986. Methods for the Determination of Organic Compounds in Finished Drinking Water and Source Water. Environmental Monitoring and Support Laboratory, Cincinnati.

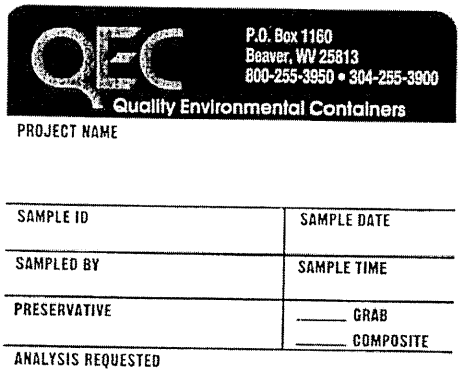
USEPA, 1986. Test Methods for Evaluating Solid Waste, SW 846, 3rd ed., vol. Ia-c, II.

National Environmental Laboratory Accreditation Conference (NELAC) Chapter 5: Quality Systems Revision 16, July 2002

APPENDIX A

Sample Label Example and Chain-of-Custody Form

Sample Label Example



CHAIN-OF-CUSTODY

Firstechnology, Inc.

4450 Johnston Parkway, Unit B
Cleveland, Ohio 44128
Tel: 216- 663-0808 • Fax: 216- 663-0656

CUSTOMER BILLING INFORMATION

COMPANY NAME _____ ADDRESS _____

CONTACT NAME _____ CITY _____

STATE/ZIP _____

PHONE ( ) _____

FAX ( ) _____

Purchase Order No. _____

SAMPLES WILL BE DISPOSED OF IN ACCORDANCE WITH FIRSTECHNOLOGY, INC.'S TERMS & CONDITIONS

COLLECTOR(S) NAME(S) print _____

COLLECTOR(S) SIGNATURE(S) _____

TURN-AROUND (circle): Regular Rush* (notify lab prior to delivery) *Needed by_____

PRIORITY SERVICE APPROVED BY:

SITE LOCATION:

DATE LOGGED: VISIBLE ICE (circle): Yes No

PRESERV.LOC. (circle) FIELD LAB NONE TEMP. (°C)

PARAMETER

FIRSTECHNOLOGY INC. #	CUSTOMER SAMPLE IDENTIFICATION	DATE COLLECTED	TIME COLLECTED	SAMPLE MATRIX	No. of CON-TAINERS											REMARKS

Relinquished by (sign)	Date/Time	Received by (sign)	Date/Time
Relinquished by (sign)	Date/Time	Received by (sign)	Date/Time
Relinquished by (sign)	Date/Time	Received for Laboratory by (sign)	Date/Time

Method of Shipment (circle)
US MAIL UPS FED. EX. FIRSTECHNOLOGY PICKUP PERSONAL DELIVERY OTHER

Preliminary Report Requested by (circle): Phone Fax E-mail

Report to be sent (if different than customer billing information):

NAME _____

ADDRESS: _____

CITY/STATE/ZIP _____

PHONE ( ) _____ FAX ( ) _____

EMAIL: _____

WHITE- LAB YELLOW - REPORT PINK - CUSTOMER

APPENDIX B

Environmental Sampling Guide

FIRSTECHNOLOGY, INC.

ENVIRONMENTAL SAMPLING GUIDE

PARAMETER	METHOD [1] SW-846 / W & W	METHOD [1] Other	CONTAINER [2]	PRESERVATION	MAXIMUM HOLDING TIME	SAMPLE REQUIRED Weight/Volume
BACTERIOLOGICAL TESTS:						
Coliform, fecal	SM9222 D [3]		P,G	0.008 % Sodium thiosulfate	6 hours	--- 200 ml
total, potable	SM9221 D [3]		P,G	0.008 % Sodium thiosulfate	30 hours	--- 200 ml
total, non-potable	SM9221 D [3]		P,G	0.008 % Sodium thiosulfate	6 hours	--- 200 ml
Standard plate count	SM9215 [3]		P,G	0.008 % Sodium thiosulfate	6 hours	--- 200 ml
Streptococci, fecal	SM9230 C [3]		P,G	0.008 % Sodium thiosulfate	6 hours	--- 200 ml
INORGANIC & PHYSICAL TESTS:						
Acidity	----	305.2, SM2310 B [3]	P,G	Cool, 4 deg. C	14 days	--- 100 ml [15]
Alkalinity	----	310.1, SM2320 [3]	P,G	Cool, 4 deg. C	14 days	--- 100 ml [15]
Biochemical Oxygen Demand (BOD)	SM5210 B [3], 405.1		P,G	Cool, 4 deg. C	48 hours	--- 1 liter
Carbonaceous BOD	----	405.1	P,G	Cool, 4 deg. C	48 hours	--- 1 liter
Boron	6010, 6020		P only	Nitric acid to pH<2	6 months	10 g 250 ml
Bromide	4500 Br-B [3]		P,G	None required	28 days	--- 100 ml
Caloric content	ASTM D240-87		P,G	None required	Not applicable	50 g 50 ml
Carbon, total organic (TOC)	9060	415.2	G only	[4]	28 days	10 g 25 ml
Chemical oxygen Demand (COD)		SM 5220 [3], 410.4	P,G	[4]	28 days	--- 50 ml
Chloride	9251	325.2	P,G	None required	28 days	10 g 50 ml
Chlorine, residual	SM4500 Cl-G [3]		P,G	None required	Analyze Immed.	--- 200 ml
Chlorine by bomb	ASTM 2361-85		G	None required	Not applicable	50 g 50 ml
Color	2120B[3]	110.3	P,G	Cool, 4 deg. C	48 hours	--- 50 ml
Conductivity	9050	120.1, SM2510 B [3]	P,G	Cool, 4 deg. C	28 days	100 g 100 ml

FIRSTECHNOLOGY, INC.

ENVIRONMENTAL SAMPLING GUIDE

PARAMETER	METHOD [1] SW-846 / W & W	METHOD [1] Other	CONTAINER [2]	PRESERVATION	MAXIMUM HOLDING TIME	SAMPLE REQUIRED Weight/Volume
Cyanide, total	9012	335.2	P,G	NaOH to pH=12, 0.6g	14 days	10 g 500 ml
amenable		335.1		Ascorbic acid [5]		
free	SM 4500 CN- I [3]					
reactive	7.3.3.2 (9010)					
Fluoride	----	340.2, SM4500-F C [3]	P,G	None required	28 days	--- 100 ml
Formaldehyde	NIOSH 3500M		G	Sodium Bisulfite	30 days	2 - 40 ml Vials
Halogen, organic	9020	-----	P,G	Cool, 4 deg. C	7 days	10 g -----
extractable (EOX)	9076					
by Bomb	5050					
Hardness	6010	SM2340 B [3], 200.7	P,G	Nitric acid to pH<2	6 months	--- 100 ml
Ignitability (flashpoint)	1010	-----	P,G	None required	Not applicable	50 g 100 ml
MBAS	----	425.1, SM5540 C [3]	P,G	Cool, 4 deg. C	48 hours	--- 250 ml
Nitrogen:						
Ammonia	----	350.1, SM4500-NH3 H [3]	P,G	[4]	28 days	50 g 500 ml
Kjeldahl (TKN)	----	351.1, SM4500-Norg [3]	P,G	[4]	28 days	50 g 500 ml
Nitrate	----	353.2, 353.1, SM4500-NO3 F [3]	P,G	Cool, 4 deg. C	48 hours	--- 100 ml
Nitrite	----	353.2, 353.1, SM4500-NO3 F [3]	P,G	Cool, 4 deg. C	48 hours	10 g 100 ml
Nitrate + Nitrite	----	353.2, 353.1, SM4500-NO3 F [3]	P,G	[4]	28 days	10 g 100 ml
Organic, total (TON)	SM4500-N org. B [3]		P,G	[4]	28 days	50 g 500 ml
Oil & Grease		1664	G only	[7]	28 days	10 g 1 liter
Oxygen, dissolved						
probe	----	360.1	BOD bottle	None required	Analyze Immed.	--- 300 ml

FIRSTECHNOLOGY, INC.

ENVIRONMENTAL SAMPLING GUIDE

PARAMETER	METHOD [1] SW-846	METHOD [1] Other	CONTAINER [2]	PRESERVATION	MAXIMUM HOLDING TIME	SAMPLE REQUIRED Weight	SAMPLE REQUIRED Volume
Paint filter	9095	-----	P,G	None required	14 days	200 g	----
Petroleum hydrocarbon, total recoverable (TPH)		1664	G only	[7]	28 days	10 g	1 liter
pH (hydrogen ion)	9040,9045	150.1, SM4500-H B [3]	P,G	None required	Analyze Immed.	100 g	25 ml
Phenolics	9065	420.1	G only	[4]	28 days	10 g	1 liter
Phosphate, total	----	365.1, SM4500-P F [3]	P,G	[4]	28 days	25 g	100 ml
Phosphate, ortho	----	365.1, SM4500-P F [3]	P,G	None required	48 hours	25 g	100 ml
Phosphorus, total	----	365.1, SM4500-P F [3]	P,G	[4]	28 days	25 g	100 ml
Silica	----	370.1	P only	Cool, 4 deg. C	28 days	---	50 ml
Solids (Residue):							
Filterable (TDS)	----	160.1, SM2540 C [3]	P,G	Cool, 4 deg. C	7 days	---	250 ml
Non-filterable (TSS)	----	160.2, SM2540 D [3]	P,G	Cool, 4 deg. C	7 days	---	250 ml
Total (TS)	----	160.3, SM2540 B [3]	P,G	Cool, 4 deg. C	7 days	---	250 ml
Total volatile (TVS)	----	160.4, SM2540 E [3]	P,G	Cool, 4 deg. C	7 days	---	250 ml
Settleable (SS)	----	160.5, SM2540 F [3]	P,G	Cool, 4 deg. C	48 hours	---	1 liter
Specific gravity	SM2710 F [3]		P,G	Cool, 4 deg. C	?		?
% Ash	SM2540 G [3]		P,G	Cool, 4 deg. C	7 days		50 g
Specfic gravity	SM2710 F [3]		P,G	None required	28 days	25 g	100 ml
Sulfate	9038	375.4	P,G	Cool, 4 deg. C	28 days	10 g	1000 ml
Sulfide, total	9030	376.1	P,G	NaOH to pH=12	14 days	50 g	500 ml
reactive	7.3.4.2(9034)						
Sulfite	4500-SO3 [3]		P,G	None required	48 hours	25 g	250 ml
Temperature	----	170.1	P,G	None required	Analyze Immed.	---	100 ml
Turbidity	----	180.1	P,G	Cool, 4 deg. C	48 hours	---	500 ml

FIRSTECHNOLOGY, INC.

ENVIRONMENTAL SAMPLING GUIDE

PARAMETER	METHOD [1] SW-846 / W & W	METHOD [1] Other	CONTAINER [2]	PRESERVATION	MAXIMUM HOLDING TIME	SAMPLE REQUIRED Weight	SAMPLE REQUIRED Volume
METALS:							
Chromium VI (wet chemical test)	7196	3500-Cr D [3]	P,G	Cool, 4 deg. C	24 hours	50 g	250 ml
Mercury							
waste & wastewater	6010, 7470, 7471		P,G	Nitric acid to pH<2	28 days [8]	10 g	250 ml
drinking water		245.1	G only	Nitric acid to pH<2	28 days [8]	---	250 ml
low-level	1631		G only	BrCl 5mL/L	28 days		500 ml
Metals except Cr VI & Hg	6010-7000 series	200 series	P,G	Nitric acid to pH<2	6 months [8]	10 g	250 ml
ORGANIC ANALYSES:							
Base/Neutrals, Acids, PAHs extractables [9]			G only w/TFE cap	Cool, 4 deg. C 0.008% Sodium thiosulfate [5]	[8] [10]		
solids, sludges, oils	8270					60 g	
water/wastewater	8270	625					2 liters
Herbicides, chlorinated			G only w/TFE cap	Cool, 4 deg. C 0.008% Sodium thiosulfate [5]	[8] [10]		
solids, sludges, oils	8151					60 g	
wastewater	8151,	1978					
drinking water		515.1					2 liters
Pesticides, chlorinated			G only w/TFE cap	Cool, 4 deg. C, pH 5-9 0.008% Sodium thiosulfate [5]	[8] [10]		
solids, sludges, oils	8081					60 g	
water/wastewater	8081	608					2 liters
drinking water		508					2 liters
Polychlorinated biphenyls (PCBs)			G only w/TFE cap	Cool, 4 deg. C 0.008% Sodium thiosulfate [5]	[10]		
solids, sludges, oils	8082					60 g	
water/wastewater	8082	608					2 liters

ESGuide9.XLS

FIRSTECHNOLOGY, INC.

ENVIRONMENTAL SAMPLING GUIDE

PARAMETER	METHOD [1] SW-846 / W & W	METHOD [1] Other	CONTAINER [2]	PRESERVATION	MAXIMUM HOLDING TIME	SAMPLE REQUIRED Weight/Volume
Volatile organic compounds (VOCs) [11]:						
solids, sludges, oils GC/MS	8260		G only w/TFE cap	Cool, 4 deg. C	14 days [8]	10 g [13]
water/wastewater GC/MS	8260	624	VOA vial w/TFE septum	Cool, 4 deg. C Hydrochloric acid to pH<2	14 days	2 X 40 ml
soils GC/MS	8260		Encore Sampler or VOA vial w/disposable syringe	Cool, 4 deg. C 1 vial: 1g sodium bisulfate; 1 vial: 10 mL of methanol	14 days	3 X 40 ml
drinking water		502.2 524.2	VOA vial w/TFE septum	Cool, 4 deg. C Hydrochloric acid to pH<2 25 mg. Ascorbic acid [5]	14 days	2 X 40 ml
BTEX only[14], GC/MS						
solids, sludges, oils	8260		G only w/TFE cap	Cool, 4 deg. C	14 days	10 g [13]
water/wastewater	8260	624	VOA vial w/TFE septum	Cool, 4 deg. C	14 days	2 X 40 ml
Total Petroleum Hydrocarbon (TPH) GC/FID	8015M					
aqueous liquid			VOA vial w/TFE septum	Cool, 4 deg. C	14 days	1 Liter 2 X 40 ml
solids, sludges, oils			G only w/TFE cap	Cool, 4 deg. C	14 days	10 g [13]
Trihalomethanes only (THMs)						
wastewater drinking water		624 524.2	VOA vial w/TFE septum	Cool, 4 deg. C	14 days	2 X 40 ml

FIRSTECHNOLOGY, INC.

ENVIRONMENTAL SAMPLING GUIDE

PARAMETER	METHOD [1] SW-846 / W & W Other	CONTAINER [2]	PRESERVATION	MAXIMUM HOLDING TIME	SAMPLE REQUIRED Weight/Volume

[1]-EPA SW-846 Solid Waste Methods, EPA Series 100-600 Methods for Chemical Analysis of Water/Wastewater
[2]-P=High density polyethylene, G=Glass
[3]-Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF, 18 Ed., 1989.
[4]-Cool, 4 deg. C, sulfuric acid to pH<2
[5]-To be used only in the presence of residual chlorine
[6]-Solid waste extraction method
[7]-Cool, 4 deg. C, hydrochloric or sulfuric acid to pH<2
[8]-Holding time until TCLP extraction is 28 days for mercury, 6 mos. for other metals and 14 days for organic analytes.
[9]-Includes benzidines, phthalates, PAHs, phenols and all other semi-volatiles included in Priority Pollutants, Hazardous Substance List, Appendix IX, Target Compound List, and Toxic Characteristic Leachate Procedure
[10]-For wastes: 7 days until extraction, 40 days after extraction. For liquids 7 days until extraction, 40 days after extraction. For drinking water: 7 days until extraction, 14 days after extraction
[11]-Includes all VOC analytes in Priority Pollutants, Hazardous Substance List, Appendix IX, Target Compound List and Toxic Characteristic Leachate Procedure.
[12]-Requests for full TCLP require 200 grams per sample.
[13]-Fill container completely to minimize headspace.
[14]-Benzene, Toluene, Ethyl benzene, Xylenes (major gasoline constituents).

APPENDIX C

Examples of Internal Analytical Result Report Forms

PARAMETER:

Reagent Lot #s:

Standard Lot #s:

Primary:

Secondary:

METHOD:

	FIRSTECH #	BOTTLE ID	DATE	MATRIX	INITIAL WT./VOL.	DILUTION	INSTRUMENT READING/ TITRANT VOL.	SAMPLE RESULT CALCULATION *	
1									1
2									2
3									3
4									4
5									5
6									6
7									7
8									8
9									9
10									10
11									11
12									12
13									13
14									14
15									15
16									16

*SHOW CALCULATION AT LEAST ONCE FOR EACH SAMPLE BATCH OR SET

PARAMETER:

Spec Wavelength: _____ nm

ACCEPTANCE LIMITS:

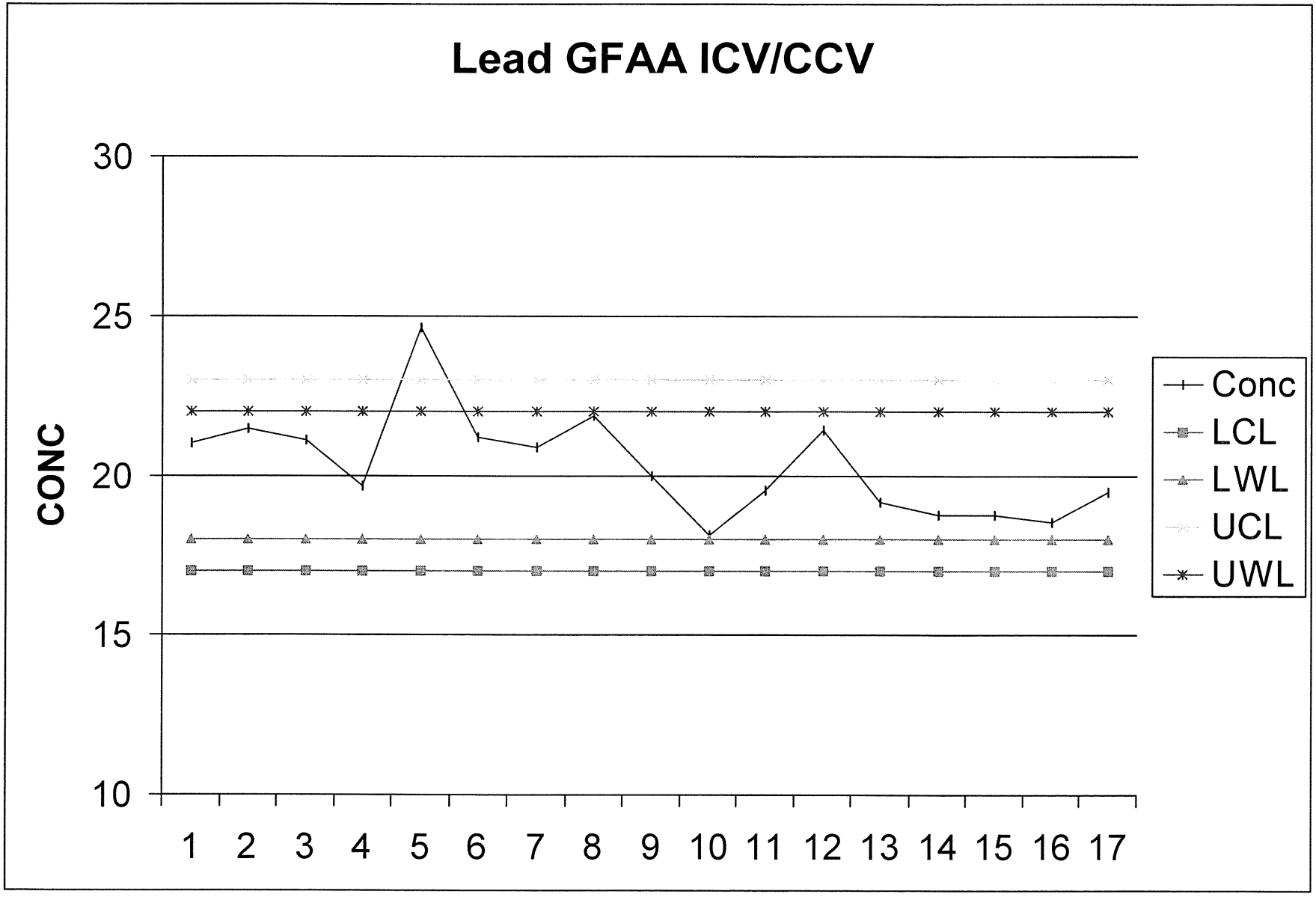
Blanks: <Detection Limit
LCS: 85 - 115%
Dups: 80 - 120%
Prim./Sec Stds: 90 - 110%
Spikes (Non-DW): 75 - 125%
Spikes (DW): 90 - 110%

FINAL RESULT	DETECTION LIMIT	SPIKE/STD CONCENTRATION *	SPIKE/STD RECOVERY *	INITIALS

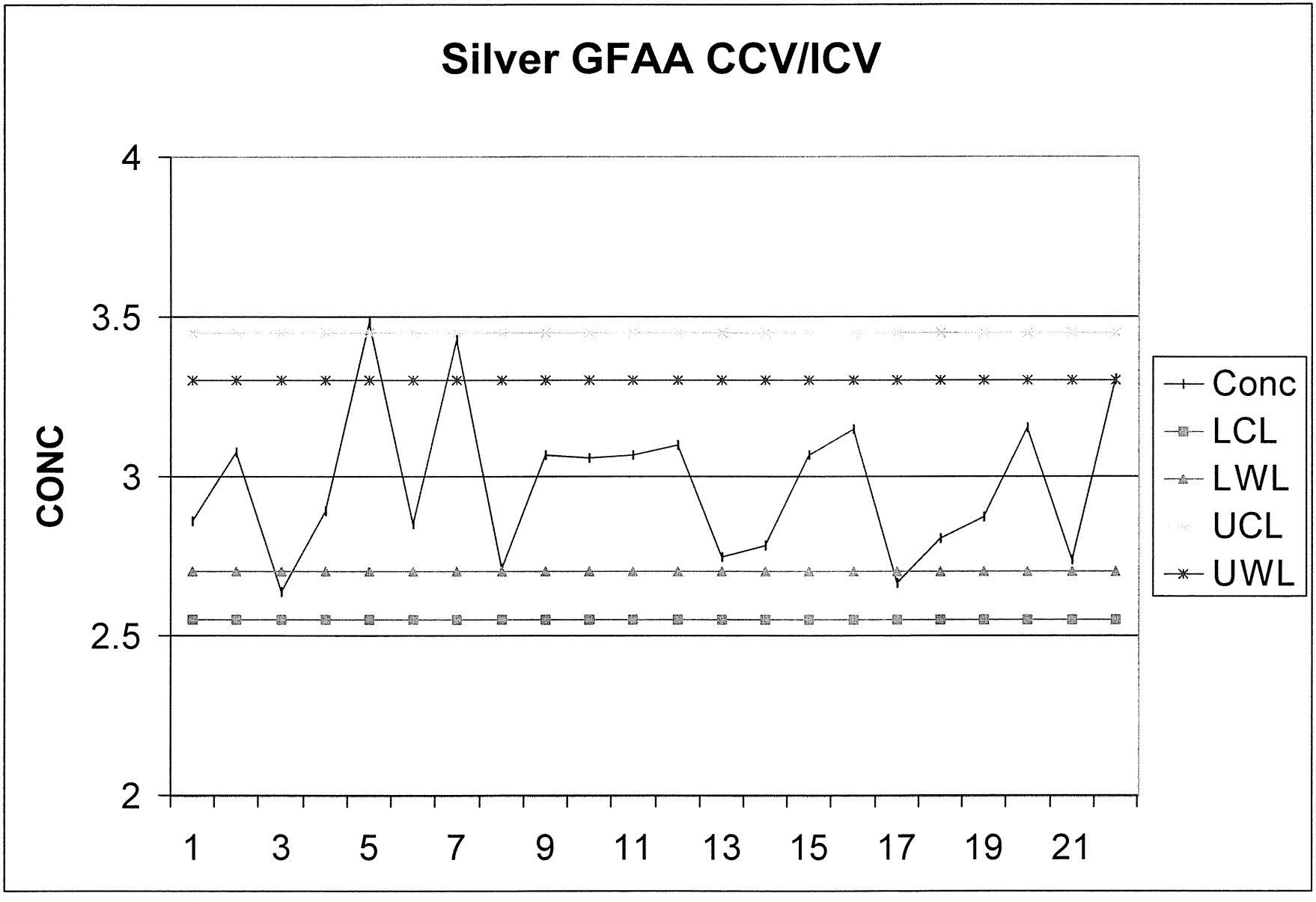
*SHOW CALCULATION AT LEAST ONCE FOR EACH SAMPLE BATCH OR SET

APPENDIX D

Examples of Statistical Quality Control Charts


Lead GFAA ICV/CCV
CONC
30
25
20
15
10
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Conc
LCL
LWL
UCL
UWL

10/6/05


Silver GFAA CCV/ICV
CONC
4
3.5
3
2.5
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1
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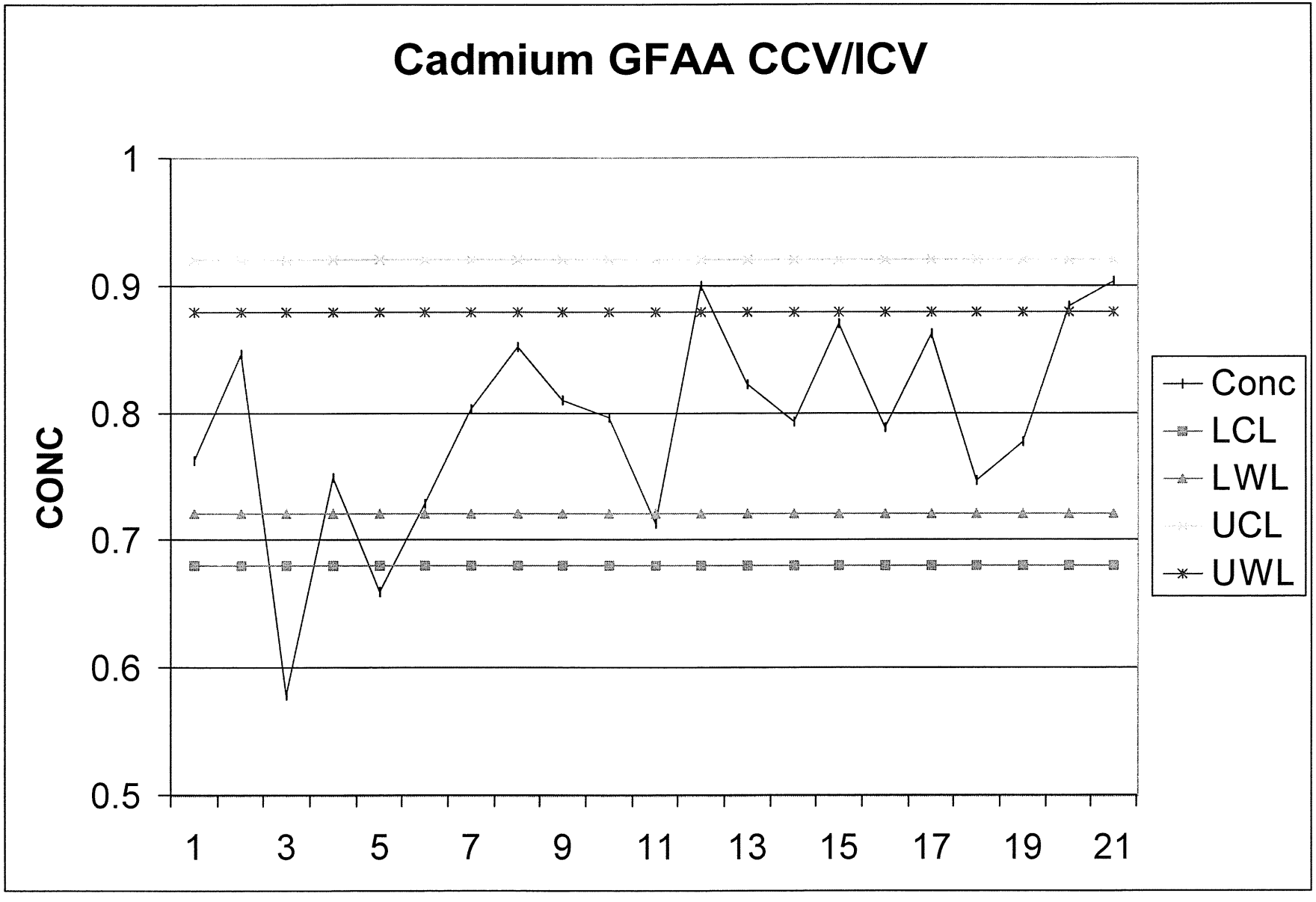
Conc
LCL
LWL
UCL
UWL

10/6/05


Cadmium GFAA CCV/ICV
CONC
1
0.9
0.8
0.7
0.6
0.5
1
3
5
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13
15
17
19
21
Conc
LCL
LWL
UCL
UWL

10/6/05

APPENDIX E

Examples of Final Analytical Report

FIRSTECHNOLOGY, Inc.
4450 Johnston Parkway
Cleveland, OH 44128

Phone: (216) 663-0808
Fax: (216) 663-0656
E-Mail: firstlabs@sbcglobal.net

Report of Analysis

Name: Attn: Philip Byron
Firstech, Inc.
19701 South Miles Rd.
Warrensville Heights, OH 44128
Sample Date: 1/30/2006
Sample Time: 3:00:00 PM
Receipt Date: 1/30/2006 4:00:00 PM
Report Date: 5/5/2006
Sample Site:

Sample ID# 84966
Sample Type: Liquid
Sample Source:
Sampler's Name: MAK
Client Sample ID: LIQ 013006

Parameter	Sample Result	Units	Limit	Minimum Detection Level	Analysis Date	Analyst
Inorganic Group						
Ignitability	85	F	140	32	2/10/2006	WAM
Reactive Cyanide	ND	mg/Kg	No limit set	0.5	2/2/2006	WAM
Reactive Sulfide	ND	mg/Kg	No limit set	50	2/3/2006	WAM
Physical						
pH	< 1	SU	2 - 12.5	0	2/3/2006	AW
TCLP Metals						
Arsenic (TCLP)	ND	mg/L	5	1	2/13/2006	SCB
Barium (TCLP)	ND	mg/L	100	2	2/13/2006	SCB
Cadmium (TCLP)	2.18	mg/L	1	0.1	2/13/2006	SCB
Chromium (TCLP)	1000	mg/L	5	10	2/13/2006	SCB
Lead (TCLP)	3.46	mg/L	5	0.2	2/13/2006	SCB
Mercury (TCLP)	4.50	mg/L	0.2	3	2/8/2006	RLG
Selenium (TCLP)	ND	mg/L	1	1	2/13/2006	SCB
Silver (TCLP)	ND	mg/L	5	0.1	2/13/2006	SCB

ND = Not Detected

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Report Date: 5/5/2006
Sample Site:

Sample ID# 84966
Sample Type: Liquid
Sample Source:
Sampler's Name: MAK
Client Sample ID: LIQ 013006

Parameter	Sample Result	Units	Limit	Minimum Detection Level	Analysis Date	Analyst
TCLP Organics						
1,1-Dichloroethene	ND	mg/L	0.7	24	2/7/2006	AC
1,2-Dichloroethane	ND	mg/L	0.5	24	2/7/2006	AC
1,4-Dichlorobenzene	ND	mg/L	7.5	24	2/7/2006	AC
2,4,5-TP (Silvex)	ND	mg/L	1	0.0005	2/14/2006	PG
2,4,5-Trichlorophenol	ND	mg/L	400	0.4	2/15/2006	AC
2,4,6-Trichlorophenol	ND	mg/L	2	0.4	2/15/2006	AC
2,4-D	ND	mg/L	10	0.005	2/14/2006	PG
2,4-Dinitrotoluene	ND	mg/L	0.13	0.4	2/15/2006	AC
2-Butanone (MEK)	ND	mg/L	200	240	2/7/2006	AC
2-Methylphenol	ND	mg/L	200	0.4	2/15/2006	AC
3&4-Methylphenol	ND	mg/L	200	0.8	2/15/2006	AC
Benzene	ND	mg/L	0.5	24	2/7/2006	AC
Carbon Tetrachloride	ND	mg/L	0.5	24	2/7/2006	AC
Chlordane	ND	mg/L	0.03	0.1	2/14/2006	PG
Chlorobenzene	ND	mg/L	100	24	2/7/2006	AC
Chloroform	390	mg/L	6	24	2/7/2006	AC
Endrin	ND	mg/L	0.02	0.05	2/14/2006	PG
Heptachlor	ND	mg/L	0.008	0.02	2/14/2006	PG
Heptachlor Epoxide	ND	mg/L	0.008	0.02	2/14/2006	PG
Hexachlorobenzene	ND	mg/L	0.13	0.4	2/15/2006	AC
Hexachlorobutadiene	ND	mg/L	0.5	0.4	2/15/2006	AC
Hexachloroethane	ND	mg/L	3	0.4	2/15/2006	AC
Lindane	ND	mg/L	0.4	0.05	2/14/2006	PG
Methoxychlor	ND	mg/L	10	0.2	2/14/2006	PG
Nitrobenzene	ND	mg/L	2	0.4	2/15/2006	AC
Pentachlorophenol	ND	mg/L	100	2	2/15/2006	AC

ND = Not Detected

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Report of Analysis

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Sample Time: 3:00:00 PM
Receipt Date: 1/30/2006 4:00:00 PM
Report Date: 5/5/2006
Sample Site:

Sample ID# 84966
Sample Type: Liquid
Sample Source:
Sampler's Name: MAK
Client Sample ID: LIQ 013006

Parameter	Sample Result	Units	Limit	Minimum Detection Level	Analysis Date	Analyst
TCLP Organics						
Pyridine	2450	mg/L	5	400	2/21/2006	AC
Tetrachloroethene	ND	mg/L	0.7	24	2/7/2006	AC
Toxaphene	ND	mg/L	0.5	1	2/14/2006	PG
Trichloroethene	ND	mg/L	0.5	24	2/7/2006	AC
Vinyl Chloride	ND	mg/L	0.2	24	2/7/2006	AC

Mark Kalmeyer, Lab Director _____

ND = Not Detected

Monique Posner, Ph.D., QA/QC Officer _____

Comment: Detection Limits for TCLP Organics were raised due to sample matrix.

Methods: Ignitability - SW846(1010): Reactive Cyanide - SW7.3.3.2(9012): Reactive Sulfide - SW7.3.4.2(9034): pH - SW846(9040): TCLP Metals - SW846(1311,6010,7470): TCLP Organics - SW846(1311,8260,8270,8081,8151)

APPENDIX F

Methods and Method Detection Limits

Detection Limit Guide

Inorganics

Parameter	Method	Liquid		Solid
		R.L. [1] mg/L	MDL [2] mg/L	R.L. [1] mg/kg
Acidity	305.1/SM2310B	10	3.0	
Alkalinity	310.1 / SM2320	10	3.0	
Ammonia as N	350.1 / SM4500-NH3 H	0.2	0.18	
%Ash, Gravimetric	SM2540 G			0.01%
%Ash, by Bomb	ASTM D240-87			0.01%
ASTM Leaching Procedure	ASTM D3987-85	N/A	N/A	
Biochemical Oxygen Demand (BOD)	405.1	2	0.68	
Carbon, Total Organic (TOC)	415.2	1	0.30	
Carbonaceous BOD	405.1	2	0.68	
Chemical Oxygen Demand	410.4 / SM5220	10	4.4	
Chloride	325.2 / SW846(9251)	3	2.6	
Chromium, Hexavalent	SM3500-Cr D	0.01	0.0025	
Conductivity (Specific Conductance)	120.1 / SM2510 B / SW846 (9050)			
Cyanide, Total	335.2 / SW846(9012)	0.01	0.008	0.5
Cyanide, Amenable	335.1 / SM4500-CN G	0.01	0.008	0.5
Cyanide, Free	SM4500-CN I	0.01	0.008	0.5
Cyanide, Reactive	SW846-7.3.3.2(9010)			0.5
Fluoride	340.2 / SM4500-F C	0.1	0.033	
Formaldehyde	NIOSH 3500M	0.1		0.25
Halogen, Total Organic (TOX) (water)	SW846(9020)	0.01		
Halogen, Total Organic (TOX) (oil)	SW846(9076)			10
Hardness	200.7 / SM2340 B / SW846(6010)	See metals	See metals	
Ignitability (Flashpoint)	SW846 (1010)	25°C	N/A	
MBAS (Surfactants)	425.1 / SM5540 C	0.1	0.078	
Nitrate + Nitrite as N or Individually	353.2 / SM4500-NO3 F	0.05	0.042	
Nitrogen, Total Kjeldahl (TKN)	351.3 / SM4500-Norg	0.2	0.18	
Nitrogen, Total Organic (TON)	SM4500-Norg	0.2	0.18	
Oil & Grease	1664	5	1.1	
Oxygen, Dissolved (Probe)	360.1	2	-	
Paint Filter (Free Liquids)	SW846 (9070)	Pass/Fail	N/A	
Petroleum Hydrocarbons, total (TPH)	1664	5	1.1	10
pH (Hydrogen Ion), water	EPA 150.1 / SM4500-H B / SW846(9040)	N/A	N/A	
pH (Hydrogen Ion), solid, oil	SW846(9045)	N/A	N/A	
Phenolics	420.1 / SW846(9065)	0.05	0.017	
Phosphate, ortho	365.1 / SM4500-P F	0.02	0.014	
Phosphate, total	365.1 / SM4500-P F	0.02	0.014	
Phosphorous, total	365.1 / SM4500-P F	0.02	0.014	
Solids, Total Dissolved (TDS)	160.1 / SW2540 C	10 [3]	--	

Detection Limit Guide

Inorganics

Parameter	Method	Liquid		Solid
		R.L. [1] mg/L	MDL [2] mg/L	R.L. [1] mg/kg
Solids, Non-Filterable (TSS)	160.2 / SM2540 D	4 [3]	--	
Solids, Total (TS)	160.3 / SM2540 B	10 [3]	--	
Solids, Fixed and Volatile (TVS/TVSS) (water)	160.4 / SM2540 E	10 [3]	--	
Solids, Fixed and Volatile (TVS/TVSS) (solid)	SM2540 G			0.01%
Solids, Settleable (SS)	160.5 / SM2540 F	0.20 mL/L/hr		
Specific Gravity	SM2710 F	N/A	N/A	
Sulfate	375.4 / SW846(9038)	5	1.1	
Sulfide, Total	376.1 / SW846(9030)	2	0.16	
Sulfide, Reactive	SW846-7.3.4.2(9034)			50
Sulfite	SM4500-SO3	2	0.16	
Sulfur	ASTM D129-64			
TCLP Leaching Procedure	SW846(1311)	N/A	N/A	
Turbidity	180.1 / SM2130 B	0.4	0.36	

Notes:

[1] Reporting Limits will vary degending upon sample size/volume, dilution factors and MDLs.

[2] MDLs are determined on an annual basis and are subject to change.

[3] TDS Reporting Limit is based on sensitivity of analytical balance used for analysis.

Detection Limit Guide

Reporting Limits for Metals & Minerals

Element	Method	MDL [2] Liquid ug/L	Liquid R.L. [1] ug/L	Solid R.L. [3] mg/kg
Aluminum	SW846(6010) / 200.7	21	100	5
	SW846(6020) / 200.8	0.14	1	0.05
Antimony	SW846(6010) / 200.7	26	100	5
	SW846(6020) / 200.8	0.044	1	0.05
	SW846(7041) / 204.2*		2	0.1
Arsenic	SW846(6010) / 200.7	45	100	5
	SW846(6020) / 200.8	0.22	1	0.05
	SW846(7060) / 206.2*		2	0.1
Barium	SW846(6010) / 200.7	8.0	5	0.25
	SW846(6020) / 200.8	0.057	0.4	0.02
Beryllium	SW846(6010) / 200.7	0.92	10	0.5
	SW846(6020) / 200.8	0.053	0.2	0.01
	SW846(7091) / 210.2*		0.5	0.025
Boron	SW846(6010) / 200.7	2.6	10	0.5
Cadmium	SW846(6010) / 200.7	4.9	10	0.5
	SW846(6020) / 200.8	0.034	0.4	0.02
	SW846(7131) / 213.2*		0.2	0.01
Chromium	SW846(6010) / 200.7	4.8	10	0.5
	SW846(6020) / 200.8	0.028	0.4	0.02
Cobalt	SW846(6010) / 200.7	9.1	10	0.5
	SW846(6020) / 200.8	0.03	1	0.05
Copper	SW846(6010) / 200.7	4.4	10	0.5
	SW846(6020) / 200.8	0.11	0.4	0.02
Gold	SW846(6010) / 200.7			
Lead	SW846(6010) / 200.7	5.2	20	1
	SW846(6020) / 200.8	1.0	0.4	0.02
	SW846(7421) / 239.2*		2	0.1
Lithium	SW846(6010) / 200.7			
	SW846(6020) / 200.8			
Mercury	SW846(7470) / SW846(7471) / 245.1	0.13	0.2	0.01
Molybdenum	SW846(6010) / 200.7	14	20	1
	SW846(6020) / 200.8	0.72	1	0.05
Nickel	SW846(6010) / 200.7	6.8	10	0.5
	SW846(6020) / 200.8	0.031	0.4	0.02
Niobium	SW846(6010) / 200.7			
Palladium	SW846(6010) / 200.7			
Platinum	SW846(6010) / 200.7			
Ruthenium	SW846(6010) / 200.7			

Detection Limit Guide

Reporting Limits for Metals & Minerals

Element	Method	MDL [2] Liquid ug/L	Liquid R.L. [1] ug/L	Solid R.L. [3] mg/kg
Selenium	SW846(6010) / 200.7	44	100	5
	SW846(6020) / 200.8	0.34	1	0.05
	SW846(7740) / 272.2*		2	0.1
Silver	SW846(6010) / 200.7	4.3	10	0.5
	SW846(6020) / 200.8	0.042	0.4	0.02
	SW846(7761) / 272.2*		0.5	0.025
Strontium	SW846(6010) / 200.7	0.88	10	0.5
Tellurium	SW846(6010) / 200.7			
Thallium	SW846(6010) / 200.7	42	100	5
	SW846(6010) / 200.7	0.031	0.4	0.02
	SW846(7841) / 279.2*		2	0.1
Tin	SW846(6010) / 200.7	37	100	5
Titanium	SW846(6010) / 200.7	3.3	5	0.25
Tungsten	SW846(6010) / 200.7			
Vanadium	SW846(6010) / 200.7	4.6	10	0.5
	SW846(6020) / 200.8	0.39	1	0.05
Zinc	SW846(6010) / 200.7	4.5	10	0.5
	SW846(6020) / 200.8	0.81	1	0.05
Zirconium	SW846(6010) / 200.7			
Calcium	SW846(6010) / 200.7	1.3	50	2.5
Magnesium	SW846(6010) / 200.7	2.4	20	1
Potassium	SW846(6010) / 200.7	12	20	1
Sodium	SW846(6010) / 200.7	11	20	1
Iron	SW846(6010) / 200.7	13	20	1
Manganese	SW846(6010) / 200.7	2.2	10	0.5
	SW846(6020) / 200.8	0.26	1	0.05

Notes:

References for Digestion Methods:

Water - ICP: SW-846 3010; Hg: SW-846 7470

Soil - ICP: SW-846 3050; Hg: SW-846 7471

[1] Reporting Limits will vary degending upon sample size/volume, dilution factors, MDLs, and dry weight reporting for soil samples.

[2] MDLs are determined on an annual basis and are subject to change.

[3] Solid Limits are calculated based on liquid MDL using standard soil dilution factor (1 gram to 50 mL.)

* Reporting Limts for GFAA analyses are based on lowest calibration standard

Detection Limit Guide

Reporting Limits for Volatile Organic Compounds
EPA SW-846 (8260), EPA 624

Compound	Liquid		Solid	
	R.L. [1] ug/L	MDL [2] ug/L	R.L. [1] ug/kg	MDL [2] ug/kg
1,1,1,2-Tetrachloroethane	5.0	1.92	5.0	1.92
1,1,1-Trichloroethane	5.0	2.62	5.0	2.62
1,1,2,2-Tetrachloroethane	5.0	3.33	5.0	3.33
1,1,2-Trichloroethane	5.0	3.32	5.0	3.32
1,1-Dichloroethane	5.0	2.80	5.0	2.80
1,1-Dichloroethene	5.0	3.28	5.0	3.28
1,1-Dichloropropene	5.0	1.87	5.0	1.87
1,2,3-Trichloropropane	5.0	1.73	5.0	1.73
1,2,4-Trimethylbenzene	5.0	2.03	5.0	2.03
1,2-Dibromo-3-chloropropane	20	17.5	20	17.5
1,2-Dibromoethane	5.0	2.40	5.0	2.40
1,2-Dichlorobenzene	5.0	2.61	5.0	2.61
1,2-Dichloroethane	5.0	2.75	5.0	2.75
1,2-Dichloropropane	5.0	1.52	5.0	1.52
1,3,5-Trimethylbenzene	5.0	1.83	5.0	1.83
1,3-Dichlorobenzene	5.0	1.78	5.0	1.78
1,4-Dichlorobenzene	5.0	1.32	5.0	1.32
1,4-Dioxane	100	23.2	100	23.2
2,2-Dichloropropane	5.0	2.52	5.0	2.52
2-Butanone (MEK)	50	5.74	50	5.74
2-Chloro-1,3-Butadiene (Chloroprene)	10	2.87	10	2.87
2-Chloroethyl Vinyl Ether	10.0	6.37	10.0	6.37
2-Chlorotoluene	5	2.47	5	2.47
2-Hexanone	5.0	6.06	5.0	6.06
2-Nitropropane	5.0	1.07	5.0	1.07
4-Chlorotoluene	5.0	2.26	5.0	2.26
4-Methyl-2-pentanone (MIBK)	50	3.39	50	3.39
Acetone	100	6.86	100	6.86
Acetonitrile	20	3.03	20	3.03
Acrolein	20	9.55	20	9.55
Acrylonitrile	20	3.24	20	3.24
Allyl Chloride (3-Chloropropene)	5.0	3.87	5.0	3.87
Benzene	5.0	2.15	5.0	2.15
Bromobenzene	5.0	1.45	5.0	1.45
Bromochloromethane	5.0	3.19	5.0	3.19

Detection Limit Guide

Reporting Limits for Volatile Organic Compounds
EPA SW-846 (8260), EPA 624

Compound	Liquid		Solid	
	R.L. [1] ug/L	MDL [2] ug/L	R.L. [1] ug/kg	MDL [2] ug/kg
Bromodichloromethane	5.0	2.65	5.0	2.65
Bromoform	5.0	1.89	5.0	1.89
Bromomethane	10	1.63	10	1.63
Carbon Disulfide	5.0	5.28	5.0	5.28
Carbon Tetrachloride	5.0	2.15	5.0	2.15
Chlorobenzene	5.0	1.44	5.0	1.44
Chloroethane	10	2.87	10	2.87
Chloroform	5.0	2.43	5.0	2.43
Chloroiodomethane	5.0	3.46	5.0	3.46
Chloromethane	10	3.03	10	3.03
cis-1,2-Dichloroethene	5.0	1.95	5.0	1.95
cis-1,2-Dichloroethene	5.0	1.95	5.0	1.95
cis-1,3-Dichloropropene	5.0	2.89	5.0	2.89
cis-1,4-Dichloro-2-butene	5.0	2.90	5.0	2.90
Dibromochloromethane	5.0	2.46	5.0	2.46
Dibromomethane	5.0	2.98	5.0	2.98
Dichlorodifluoromethane	20	3.13	20	3.13
Ethyl Acetate	5.0	4.28	5.0	4.28
Ethylbenzene	5.0	1.82	5.0	1.82
Ethylmethacrylate	5.0	3.03	5.0	3.03
Freon 113	5.0	2.76	5.0	2.76
Iodomethane	10	1.90	10	1.90
Isobutanol	100	7.1	100	7.1
Isopropyl Benzene	5.0	1.83	5.0	1.83
m,p-Xylene	10	4.88	10	4.88
Methacrylonitrile	20	11.0	20	11.0
Methyl Acrylate	5.0	2.78	5.0	2.78
Methylene Chloride	5.0	5.16	5.0	5.16
Methylmethacrylate	5.0	2.34	5.0	2.34
MTBE	5.0	2.68	5.0	2.68
n-Butyl Benzene	5.0	1.48	5.0	1.48
n-Propyl Benzene	5.0	2.47	5.0	2.47
o-Xylene	5.0	1.44	5.0	1.44
Pentachloroethane	5.0	2.03	5.0	2.03
p-Isopropyltoluene	5.0	1.32	5.0	1.32
Propionitrile	20	2.15	20	2.15
sec-Butylbenzene	5.0	2.03	5.0	2.03

Detection Limit Guide

Reporting Limits for Volatile Organic Compounds
EPA SW-846 (8260), EPA 624

Compound	Liquid		Solid	
	R.L. [1] ug/L	MDL [2] ug/L	R.L. [1] ug/kg	MDL [2] ug/kg
Styrene	5.0	2.42	5.0	2.42
tert-Butylbenzene	5.0	1.32	5.0	1.32
Tetrachloroethene	5.0	2.32	5.0	2.32
Toluene	5.0	2.89	5.0	2.89
trans-1,2-Dichloroethene	5.0	2.45	5.0	2.45
trans-1,2-Dichloroethene	5.0	2.45	5.0	2.45
trans-1,3-Dichloropropene	5.0	2.42	5.0	2.42
trans-1,4-Dichoro-2-butene	5.0	3.14	5.0	3.14
Trichloroethene	5.0	2.39	5.0	2.39
Trichlorofluoromethane	5.0	2.78	5.0	2.78
Vinyl acetate	50	6.23	50	6.23
Vinyl chloride	2.0	2.97	10	2.97

Notes:

Reference: SW-846 8260 with 5030 (Liquid) and 5035 (Soil).

[1] Reporting Limits (RL) are based on 5-mL injection (Liquid) or 5-g purge (Soil)

[1] Reporting Limits (RL) will vary depending upon sample size/volume, dilution factors, MDLs, and dry weight reporting for soil samples.

[2] MDLs are determined on an annual basis and are subject to change. The RL is the lowest concentration that can be achieved within specified limits of precision and accuracy during routine laboratory operations. For many analytes the RL is selected for the lowest non-zero standard in the calibration curve. Sample RLs are highy matrix-dependent. The RLs listed are provided for guidance and may not always be achievable.

Detection Limit Guide

Reporting Limits for Semi-Volatile Organic Compounds
EPA SW-846 (8270), EPA 625

Compound	Liquid		Solid	
	R.L. [1] ug/L	MDL [2] ug/L	R.L. [1] ug/kg	MDL [2] ug/kg
1,2,4,5-Tetrachlorobenzene	10	**	330	**
1,2,4-Trichlorobenzene	10	7.0	330	233
1,2-Dichlorobenzene	10	1.7	330	56
1,2-Diphenylhydrazine	10	3.3	330	111
1,3-Dichlorobenzene	10	1.8	330	58
1,3,5-Trinitrobenzene	10	**	330	**
1,3-Dinitrobenzene	10	**	330	**
1,4-Dichlorobenzene	10	1.34	330	45
1,4-Naphthoquinone	10	**	330	**
1-Naphthylamine	10	**	330	**
2,3,4,6-Tetrachlorophenol	10	**	330	**
2,4,5-Trichlorophenol	10	2.3	330	75
2,4,6-Trichlorophenol	10	2.2	330	73
2,4-Dichlorophenol	10	2.4	330	80
2,4-Dimethylphenol	10	2.6	330	87
2,4-Dinitrophenol	50	4.9	1700	162
2,4-Dinitrotoluene	10	2.0	330	67
2,6-Dichlorophenol	10	**	330	**
2,6-Dinitrotoluene	10	3.2	330	107
2-Acetylaminofluorene	10	**	330	**
2-Chloronaphthalene	10	7.0	330	233
2-Chlorophenol	10	3.3	330	110
2-Methylnaphthalene	10	6.2	330	207
2-Methylphenol	10	3.3	330	110
2-Naphthylamine	10	**	330	**
2-Nitroaniline	10	3.9	330	130
2-Nitrophenol	10	2.8	330	93
2-Picoline	10	**	330	**
3&4-Methylphenol	20	3.3	670	110
3,3'-Dichlorobenzidine	20	4.1	670	137
3,3'-Dimethylbenzidine	10	**	330	**
3-Methylcholanthrene	10	**	330	**
3-Nitroaniline	10	6.1	330	203
4,6-Dinitro-2-methylphenol	50	2.9	1700	97
4-Aminobiphenyl	10	**	330	**

Detection Limit Guide

Reporting Limits for Semi-Volatile Organic Compounds
EPA SW-846 (8270), EPA 625

Compound	Liquid		Solid	
	R.L. [1] ug/L	MDL [2] ug/L	R.L. [1] ug/kg	MDL [2] ug/kg
4-Bromophenyl-phenylether	10	6.2	330	207
4-chloro-3-methylphenol	50	3.4	1700	113
4-Chloroaniline	10	7.5	330	249
4-Chlorophenyl-phenylether	10	7.3	330	243
4-Nitroaniline	10	6.1	330	203
4-Nitrophenol	50	1.7	1700	57
4-Nitroquinoline-1-oxide	50	**	1700	**
5-Nitro-o-toluidine	10	**	330	**
7,12-Dimethylbenz(a)anthracene	10	**	330	**
a,a-Dimethylphenethylamine	10	**	330	**
Acenaphthene	10	6.0	330	200
Acenaphthylene	10	5.3	330	177
Acetophenone	10	**	330	**
Aniline	10	5.7	330	190
Anthracene	10	3.6	330	120
Aramite	20	**	670	**
Benzidine	10	4.19	330	140
Benzo(a)anthracene	10	1.4	330	47
Benzo(a)pyrene	10	1.8	330	60
Benzo(b)fluoranthene	10	2.0	330	67
Benzo(g,h,i)perylene	10	1.5	330	50
Benzo(k)fluoranthene	10	2.0	330	67
Benzoic Acid	10	1.1	330	37
Benzyl Alcohol	10	3.1	330	103
Bis(2-chloroethoxy)methane	10	**	330	**
Bis(2-chloroethyl)ether	10	2.5	330	83
Bis(2-chloroisopropyl) ether	10	5.6	330	186
Bis(2-ethylhexyl)phthalate	10	2.6	330	87
Butylbenzylphthalate	10	1.7	330	57
Chlorobenzilate	10	**	330	**
Chrysene	10	1.3	330	43
Cyclohexanone	10	8.8	330	295
Di-n-butylphthalate	10	1.8	330	60
Di-n-octylphthalate	10	2.7	330	90
Diallate	10	**	330	**

Detection Limit Guide

Reporting Limits for Semi-Volatile Organic Compounds
EPA SW-846 (8270), EPA 625

Compound	Liquid		Solid	
	R.L. [1] ug/L	MDL [2] ug/L	R.L. [1] ug/kg	MDL [2] ug/kg
Dibenzo(a,h)anthracene	10	1.1	330	37
Dibenzofuran	10	4.5	330	150
Diethylphthalate	10	2.6	330	87
Dimethoate	10	**	330	**
Dimethylphthalate	10	3.9	330	130
Dinoseb	20	**	670	**
Diphenylamine	10	**	330	**
Disulfoton	10	**	330	**
Ethyl methanesulfonate	10	**	330	**
Famphur	20	**	670	**
Fluoranthene	10	1.9	330	63
Fluorene	10	5.2	330	173
Hexachlorobenzene	10	7.2	330	240
Hexachlorobutadiene	10	7.1	330	237
Hexachlorocyclopentadiene	10	1.8	330	60
Hexachloroethane	10	6.8	330	227
Hexachlorophene	500	**	17000	**
Hexachloropropene	10	**	330	**
Indeno(1,2,3-cd)pyrene	10	4.6	330	153
Isodrin	20	**	670	**
Isophorone	10	2.8	330	93
Isosafrole	10	**	330	**
Kepone	20	**	670	**
Methapyrilene	10	**	330	**
Methyl methanesulfonate	10	**	330	**
Methyl Parathion	10	**	330	**
n-Nitrosodi-N-butylamine	10	**	330	**
n-nitroso-di-n-propylamine	10	3.4	330	113
N-Nitrosodiethylamine	10	**	330	**
n-Nitrosodimethylamine	10	1.7	330	57
n-nitrosodiphenylamine	10	2.9	330	97
N-Nitrosomethylethylamine	10	**	330	**
N-Nitrosomorpholine	10	**	330	**
N-Nitrosopiperidine	10	**	330	**
N-Nitrosopyrrolidine	10	**	330	**
Naphthalene	10	5.8	330	193

Detection Limit Guide

Reporting Limits for Semi-Volatile Organic Compounds
EPA SW-846 (8270), EPA 625

Compound	Liquid		Solid	
	R.L. [1] ug/L	MDL [2] ug/L	R.L. [1] ug/kg	MDL [2] ug/kg
Nitrobenzene	10	4.0	330	133
o,o,o-Triethyl phosphorothioate	10	**	330	**
o-Toluidine	10	**	330	**
p-Dimethylaminoazobenzene	10	**	330	**
p-Phenylenediamine	10	**	330	**
Parathion	10	**	330	**
Pentachlorobenzene	10	**	330	**
Pentachloronitrobenzene	10	**	330	**
Pentachlorophenol	50	5.4	1700	180
Phenacetin	10	**	330	**
Phenanthrene	10	3.5	330	117
Phenol	10	2.8	330	93
Phorate	10	**	330	**
Pronamide	10	**	330	**
Pyrene	10	1.9	330	63
Pyridine	10	2.9	330	97
Safrole	10	**	330	**
Tetraethyl dithiopyrophosphate (Sulfotep)	20	**	667	**
Thionazine	10	**	330	**

Notes:

Reference: SW-846 8270 with 3510 (Liquids) and 3550 (Soils)

[1] Reporting Limits are based on 1000-mL (Liquid) sample or 30.0-g (Soil) sample extracted, 1.0 mL final extract volume, 1.0 uL injection volume and dry weight correction (for) soil samples). Reporting Limits will vary if any of these factors change.

[2] MDLs are determined on an annual basis and are subject to change. The RL is the lowest concentrat can be achieved within specified limits of precision and accuracy during routine laboratory operations. many analytes the RL is selected for the lowest non-zero standard in the calibration curve. Sample RI highy matrix-dependent. The RLs listed are provided for guidance and may not always be achievable.

**MDLs for these compounds have not been determined. Reporting limits are based on the lowest calibration standard.

Detection Limit Guide

Reporting Limits for Pesticides
SW-846(8081), EPA 608

Compound	Waters		Soils	
	R.L. [1] ug/L	MDL [2] ug/L	R.L. [1] ug/kg	MDL [2] ug/kg
4,4'-DDD	0.05	0.00866	8.3	1.4
4,4'-DDE	0.05	0.00684	8.3	1.1
4,4'-DDT	0.2	0.0674	33	11
Aldrin	0.05	0.0360	8.3	6
alpha-BHC	0.05	0.00492	8.3	0.82
beta-BHC	0.05	0.0122	8.3	2
Chlordane	0.1	0.0206	17	3.4
delta-BHC	0.05	0.0197	8.3	3.3
Dieldrin	0.05	0.0772	8.3	13
Endosulfan I	0.05	0.00446	8.3	0.74
Endosulfan II	0.05	0.00904	8.3	1.5
Endosulfan sulfate	0.2	0.00492	33	0.82
Endrin	0.05	0.0127	8.3	2.1
Endrin aldehyde	0.05	0.00708	8.3	1.2
Endrin ketone	0.05		8.3	
gamma-BHC (Lindane)	0.05	0.0266	8.3	4.4
Heptachlor	0.05	0.0338	8.3	5.6
Heptachlor Epoxide	0.05	0.00406	8.3	0.68
Isodrin	0.05		8.3	
Methoxychlor	0.2	0.0185	33	3.1
Toxaphene	1.00	0.304	170	51

Notes:

Reference: SW-846 8081 with 3510 (Liquid) and 3350 (Soil).

[1] Reporting Limits are based on 1000-mL (Liquid) sample or 30.0-g (Soil) sample extracted, 1.0 mL (Liquid) or 5.0 mL (Soils) final extract volume, 2.0 uL injection volume and dry weight correction (for soil samples). Reporting Limits will vary if any of these factors change.

[2] MDLs are determined on an annual basis and are subject to change. The RL is the lowest concentration that can be achieved within specified limits of precision and accuracy during routine laboratory operations. For many analytes the RL is selected for the lowest non-zero standard in the calibration curve. Sample RLs are highy matrix-dependent. The RLs listed are provided for guidance and may not always be achievable.

Detection Limit Guide

Reporting Limits for Herbicides
SW-846(8151), EPA 1978

Compound	Waters R.L. [1] ug/L	Soils R.L. [1] ug/kg
2,4,5-T	0.50	5.0
2,4,5-TP	0.50	5.0
2,4-D	5.0	50
2,4-DB	0.1	10
Dalapon	0.01	0.1
Dicamba	0.01	0.1
Dichloroprop	0.1	10
Dinoseb	0.05	5
MCPA	10	100
MCPP	10	100

Notes:
Reference: SW-846 8151

[1] Reporting Limits are based on 100-mL (Liquid) sample or 10.0-g (Soil) sample extracted, 5.0 mL final extract volume, 1.0 uL injection volume and dry weight correction (for) soil samples). Reporting Limits will vary if any of these factors change.

[2] MDLs are determined on an annual basis and are subject to change. The RL is the lowest concentration that can be achieved within specified limits of precision and accuracy during routine laboratory operations. For many analytes the RL is selected for the lowest non-zero standard in the calibration curve. Sample RLs are highy matrix-dependent. The RLs listed are provided for guidance and may not always be achievable.

Detection Limit Guide

Reporting Limits for PCBs
SW-846(8082), EPA 608

Compound	CAS #	Waters		Soils [3]	
		R.L. [1] ug/L	MDL [2] ug/L	R.L. [1] mg/kg	MDL [2] mg/kg
Aroclor 1016	12674-11-2	0.25	0.018	0.13	0.009
Aroclor 1221	11104-28-2	0.50	0.036	0.25	0.018
Aroclor 1232	11141-16-5	0.50	0.024	0.25	0.012
Aroclor 1242	53469-21-9	0.25	0.033	0.13	0.016
Aroclor 1248	12672-29-6	0.25	0.036	0.13	0.018
Aroclor 1254	11097-69-1	0.25	0.021	0.13	0.010
Aroclor 1260	11096-82-5	0.25	0.026	0.13	0.013

Notes:

[1] Reporting Limits will vary degending upon sample size/volume, dilution factors, MDLs and dry weight reporting for soil samples.

[2] MDLs are determined on an annual basis and are subject to change.

[3] Soil Detection limits based on 10.0 g sample extracted with a final extract volume of 5mL

Detection Limit Guide

Additional Organic Parameters

Parameter	Method	Liquid R.L. [1] mg/L	Solid R.L. [1] mg/kg
Alcohols	SW846(8015)	10-25	
Dibromoethane, 1,2 - (Ethylene Dibromide (EDB)	SW846(8011) / EPA 504.1	0.1 ug/L	
Dibromo-3-chloropropane, 1,2-(DBCP)	SW846(8011) / EPA 504.1	0.1 ug/L	
Glycols	SW846(8015)	10-50 mg/L	10-50 mg/kg
Total Petroleum Hydrocarbon-Gasoline Range	SW846(8015)	1	1
Total Petroleum Hydrocarbon-Diesel Range	SW846(8015)	0.50	17
Trihalomethanes	524.2	1 ug/L	

Notes:

[1] Reporting Limits will vary degending upon sample size/volume, dilution factors and MDLs.

Detection Limit Guide

Miscellaneous Parameters (Subbed Out)

Parameter	Method	Liquid R.L. mg/L	Solid R.L. mg/kg
Asbestos (Bulk)	EPA 600/M4-82-020		
Asbestos (Filters)	NIOSH 7400A		
Coliform, total (Presence/Absence)	SM 9221 D	1 col/100 mL	
Coliform, fecal	SM9222 D	1 col/100 mL	
Gross Alpha	EPA600/4-80-032(900) / SW846(9310)	2 pCi/L	
Gross Beta	EPA600/4-80-032(900) / SW846(9315)	3 pCi/L	
Standard Plate Count	SM 9215	1 CFU/mL	
Streptococci, fecal	SM9230 C		
Viscosity	ASTM D445	0.5 cst	
% Water	Karl Fisher		

Standard Operating Procedures

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/OA/1000

Volatile Organic Compounds by GC/MS – Capillary Column Technique

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 2

SOP Effective Date: 5/31/07

Controlled Copy on Green Paper

Copy No. _____
Approved By_____
Date _____

1.0 Methods

1.1 This SOP meets the requirements of the following methods: SW846 8260A, EPA 624.

2.0 Applicable Matrices

2.1 This method applies to a wide variety of environmental matrices, including ground water, aqueous sludges, soils, sediments, acid and caustic liquors, oils, solvents, polymers, and spent carbons and catalysts.

3.0 Detection Limits

3.1 Detection limits vary per compound; see QA/QC documentation for the latest calculated detection and reporting limits

4.0 Scope and Application

4.1 MDL Studies are performed on an annual basis; see latest QA/QC documentation which details the most recent MDL study results.

GC/MS procedure to determine the concentration of purgeable organic compounds in varied matrices (see above). The compounds listed below are routinely quantified.

		Techniques			Ions	
Analyte	CAS #[b]	Purge Trap	Direct Injection	Int. Std.	Primary	Secondary
Acetone	67-64-1	pp	a	1	58	43
Acrolein	107-02-8	a	a	1	56	55
Acrylonitrile	107-02-8	pp	a	1	53	52,51
Benzene	71-43-2	a	a	1	78	-
Bromobenzene	108-86-1	a	a	3	156	77,158
Bromochloromethane	74-97-5	a	a	1	128	49,130
Bromodichloromethane	75-27-4	a	a	2	83	85,127
Bromoform	75-25-2	a	a	3	173	175,254
Bromomethane	74-83-9	a	a	1	94	96
2-Butanone (MEK)	78-93-3	pp	a	1	72	43
n-Butylbenzene	104-51-8	a	a	4	91	92,134
sec-Butylbenzene	135-98-8	a	a	3	105	134
tert-Butylbenzene	98-06-6	a	a	3	119	91,134
Carbon Disulfide	75-15-0	pp	a	1	76	78
Carbon Tetrachloride	56-23-5	a	a	1	117	119
Chlorobenzene	108-90-7	a	a	3	112	77,114
Chlorodibromomethane	124-48-1	a	a	2	129	208, 206
Chloroethane	75-00-3	a	a	1	64	66
2-Chloroethyl vinyl ether	110-75-8	a	a	2	63	65,106
Chloroform	67-66-3	a	a	1	83	85
Chloromethane	74-87-3	a	a	1	50	52
2-Chlorotoluene	95-49-8	a	a	3	91	126
4-Chlorotoluene	106-43-4	a	a	3	91	126
1,2-Dibromo-3-chloropropane	96-12-8	pp	a	4	75	155,157
1,2-Dibromoethane	106-93-4	a	a	2	129	127
Dibromomethane	74-95-3	a	a	2	93	95,174
1,2-Dichlorobenzene	95-50-1	a	a	4	146	111,148

1,3-Dichlorobenzene	541-73-1	a	a	4	146	111,148
1,4-Dichlorobenzene	106-46-7	a	a	4	146	111,148
trans-1,4-Dichloro-2-butene	110-57-6	a	a	4	53	88,75
Dichlorodifluoromethane	75-71-8	a	a	1	85	101,87
1,1-Dichloroethane	75-34-3	a	a	1	63	65,83
1,2-Dichloroethane	107-06-2	a	a	1	62	98
1,1-Dichloroethene	75-35-4	a	a	1	96	61,63
cis-1,2-Dichloroethene	156-59-2	a	a	1	96	61,98
trans-1,2-Dichloroethene	156-60-5	a	a	1	96	61,98
1,2-Dichloropropane	78-87-5	a	a	2	63	112
1,3-Dichloropropane	142-28-9	a	a	2	76	78
2,2-Dichloropropane	594-20-7	a	a	3	77	97
1,1-Dichloropropene	563-58-6	a	a	1	75	110,77
cis-1,3-Dichloropropene	10061-01-5	a	a	2	75	77,39
trans-1,3-Dichloropropene	10061-02-6	a	a	2	75	77,39
Ethylbenzene	100-41-4	a	a	3	91	106
Hexachlorobutadiene	87-68-3	a	a	4	225	223,227
2-Hexanone	591-78-6	pp	a	2	43	58,57,100
Iodomethane	74-88-4	a	a	1	142	126,141
Isopropylbenzene	98-82-8	a	a	3	105	120
p-Isopropyltoluene	99-87-6	a	a	3	119	134,91
Methylene chloride	75-09-2	a	a	1	84	86,49
4-Methyl-2-pentanone	108-10-1	pp	a	2	100	43,58,85
Methyl-t-butyl ether	1634-04-4	a	a	1	73	57
Naphthalene	91-20-3	a	a	4	128	-
n-Propylbenzene	103-65-1	a	a	3	91	120
Styrene	100-42-5	a	a	3	104	78
1,1,1,2-Tetrachloroethane	630-20-6	a	a	3	131	133,119
1,1,2,2-Tetrachloroethane	79-34-5	a	a	3	83	131,85
Tetrachloroethene	127-18-4	a	a	2	164	129,131,166
Toluene	108-88-3	a	a	2	92	91
1,2,3-Trichlorobenzene	87-61-6	a	a	4	180	182,145
1,2,4-Trichlorobenzene	120-82-1	a	a	4	180	182,145
1,1,1-Trichloroethane	71-55-6	a	a	1	97	99,61
1,1,2-Trichloroethane	79-00-5	a	a	2	83	97,85
Trichloroethene	79-01-6	a	a	2	95	97,130,132
Trichlorofluoromethane	75-69-4	a	a	1	101	103,105
1,2,3-Trichloropropane	96-18-4	a	a	3	75	77
1,2,4-Trimethylbenzene	95-63-6	a	a	3	105	120
1,3,5-Trimethylbenzene	108-67-8	a	a	3	105	120
Vinyl acetate	108-05-4	a	a	2	43	86
Vinyl chloride	75-01-4	a	a	1	62	64
o-Xylene	95-47-6	a	a	3	106	91
m,p-Xylene	108-38-3 106-42-3	a	a	3	106	91

a Adequate response by this technique
b Chemical Abstract Services Registry Number
c Soils ug/Kg & water ug/L
pp Poor purging efficiency resulting in high MDLs

Internal Std #1: **Pentafluorobenzene**
Internal Std. #2: **1,4-Difluorobenzene**
Internal Std. #3: **Chlorobenzene-d5**
Internal Std. #4: **1,4-Dichlorobenzene-d4**

5.0 Summary

5.1 This SOP provides a purge and trap method using GC/MS techniques for the detection of volatile organic compounds.

5.2 Samples can be analyzed using direct injection (in limited applications) or purge-and-trap, Method 5030A, Volatile Organic Compounds by Purge and Trap. However, this SOP only addresses sample analysis by purge-and-trap.

5.2.1 Purged sample components are trapped in a tube containing suitable sorbent materials.

5.2.2 When purging is complete, the sorbent tube is heated and back-flushed with helium to desorb trapped sample components. The analytes are desorbed directly to a large bore capillary or cryofocussed on a capillary pre-column before being flash evaporated to either a narrow bore or wide bore capillary column for analysis.

5.2.3 Wide-bore capillary columns require a jet separator, whereas narrow bore capillary columns can be directly interfaced to the ion source.

5.3 A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved using a mass spectrometer (MS). (For screening purposes, a flame ionization detector (FID) may be used.)

5.4 Concentrations of the identified components are measured by relating the response produced for that compound to the response produced by a compound that is used as an internal standard.

6.0 Responsibilities

6.1 Analyst: Personnel experienced in the use of purge-and-trap systems and gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool. A demonstration of capability must be performed before an analyst can report data.

7.0 Definitions

7.1 IS: Internal standard, a pure analyte(s) added to a solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same solution. The internal standard must be an analyte that is not a sample component.

7.2 Surrogate: A pure analyte, which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction and is measured with the same procedures used to measure other sample components. The purpose of the surrogate is to monitor method performance with each sample.

7.3 LCS: Laboratory control sample, an aliquot of reagent water to which a known amount of method analytes are added. It is analyzed like a sample and used to determine whether the method is in control.

7.4 SSS: Secondary source standard, solution purchased from a separate supplier than the primary source standards, or from the same supplier with a different lot number. Used to validate a new calibration curve.

7.5 CCV: Continuing calibration verification, sample made up at or near the midpoint of the working calibration range. Used to verify whether the calibration curve is still valid.

7.6 MS/MSD: Matrix spike and matrix spike duplicate, an aliquot of a selected sample is spiked with a known amount of the method analytes. It is analyzed like a sample and used to determine the presence of matrix interferences.

7.7 MSDS: Material Safety Data Sheet

7.8 Blank: An aliquot of reagent water that is treated as a sample and used to determine if method analytes or other interferences are present.

7.9 GC/MS: Gas chromatograph/Mass spectrometer.

7.10 ICV: Initial Calibration Verification

8.0 Interferences

8.1 Major sources of contamination are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation.

8.2 Analyses of reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted.

8.3 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After analysis of a sample containing high concentrations of volatile organic compounds, one or more instrument blanks must be analyzed to check for cross contamination.

8.3.1 This interference may be prevented by rinsing the purging apparatus and sample syringes with portions of organic-free reagent water between samples.

8.3.2 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105° C.

8.3.3 In extreme situations, the whole purge and trap device may require dismantling and cleaning.

8.4 Screening of the samples prior to purge and trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique or by SW-846 Method 3820, Hexadecane Extraction and Screening of Purgeable Organics.

8.5 Special precautions must be taken to avoid contamination when analyzing for methylene chloride. The analytical and sample storage area must be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing must be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

8.6 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank

prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

8.7 This procedure can be used to quantitate most volatile organic compounds that have boiling points below 200° C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

9.0 Environmental Health and Safety

9.1 The toxicity or carcinogenicity of chemicals used in this procedure has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Consult the FIRSTECHNOLOGY Chemical Hygiene Plan and specific chemical MSDS sheets for health and safety information.

9.2 Glycol ethers are suspected carcinogens. All solvent handling should be done in a hood while using proper protective equipment to minimize exposure to liquid and vapor. Minimum personal protection includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

9.3 The following analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

10.0 Equipment and Supplies

10.1 VOC #1

10.1.1 GC/MS Varian Saturn 2000
- 10.1.1.1 Purging Apparatus: Tekmar LSC 3020 using Purge Trap K
- 10.1.1.2 Autosampler: Archon Autosampler
- 10.1.1.3 Column: Restek RTX-VMS 60m X 0.32 mm X 1.80 um film thickness

10.2 VOC #2

10.2.1 GC/MS Hewlett Packard 5890 Series II with model 5970 mass spec detector (MSD)
- 10.2.1.1 Purging Apparatus: Tekmar model LSC 2000 using Purge Trap K
- 10.2.1.2 Autosampler: Archon Autosampler
- 10.2.1.3 Column: Restek RTX-VMS 60m X 0.25 mm X 1.40 um film thickness

10.3 Microsyringes – 10-, 25-, 100-, 250-, 500-, and 1000-µL.

10.4 Syringes – 5-mL, gas-tight

10.5 Balances – Analytical, 0.0001 g, and top-loading, 0.1 g.

10.6 Disposable pipettes – Pasteur.

10.7 Volumetric flasks, Class A – 1-mL, 5-mL and 100-mL, with ground-glass stoppers.

10.8 Spatula – Stainless steel.

11.0 Reagents and Standards

Stock calibration solutions shall be purchased as certified solutions. Certificates must be kept on file. Stock standards must be stored at –15° C ±5° C All stock standards should be replaced after six months. Standards must be compared to the initial calibration curve and second source initial calibration verification standard. It may be necessary to replace the standards more frequently if either check exceeds 20% difference When standards are replaced, new standards must meet the same QC criteria. The following are recommended surrogates and internal standards compounds These compounds can be substituted with project specified ones.

Surrogate standards	Internal standards
toluene-d8	fluorobenzene
4-bromofluorobenzene	chlorobenzene-d5
dibromofluoromethane	1,4-dichlorobenzene-d4
1,2-dichloroethane-d4	

11.1 Organic-free reagent water All references to water in this method refer to organic-free reagent water.

11.2 Methanol, CH_3OH – purge-and-trap grade, demonstrated to be free of analytes. Store apart from other solvents.

11.3 Internal standard stock solution, dilute 1mL of 2500 ug/mL 8260 Internal Standard Mix to 10mL in methanol. This will give a final concentration of 250 ug/mL.

11.4 Multi-mix calibration stock standard, dilute the following mixture of solutions to 1mL in methanol to give a nominal concentration of 100 ug/mL. The following are standard dilutions used Concentrated stock concentrations vary per vendor Adjust preparations accordingly to achieve 100 ug/mL.

11.4.1 50 uL of 2000 ug/mL mega Mix

11.4.2 50 uL of 2000 ug/mL Gas Mix

11.4.3 40 uL of 2000 ug/mL Ketone Mix

11.4.4 100 uL of 2000 ug/mL 2-Chloroethyl-vinyl-ester

11.4.5 100 uL of 2000 ug/mL of Acetate Mix

11.4.6 20 uL of 10000 ug/mL Acrylonitrile

11.5 Surrogate stock solutions:

11.5.1 Dilute 1 mL of 2500 ug/mL 8260 Surrogate Standard Mix to 10 mL in methanol This will give a final concentration of 250 ug/mL.

11.5.2 Dilute 40 uL of 2500 ug/mL 8260 Surrogate Standard Mix to 1 mL in methanol. This will give a final concentration of 100 ug/mL.

11.6 Internal Standard + Surrogate: Dilute 1 mL of each Internal Standard and Surrogate 10 mL to give 210 ug/mL concentration.

11.7 Primary source standards, internal standards, and surrogates shall be used to prepare the initial 5-point calibration curve and CCV. Purge 5 mL water.

11.7.1 For standard #1 for the 5 ug/L standard, spike 2 uL of multi mix solution into 40mL of water.

11.7.2 For standard #2 for the 10 ug/L standard, spike 4 uL of multi-mix solution and 1 mL of surrogate mix into 40mL of water

11.7.3 For standard #3 for the 20 ug/L standard, spike 8 uL of mix and 2 uL of surrogate to 40 mL of water.

11.7.4 For standard #4 for the 50 ug/L standard, spike 20 uL of mix and 3 uL of surrogate to 40 mL of water.

11.7.5 For standard #5 for the 100 ug/L standard, spike 40 uL of mix and 4 uL of surrogate to 40 mL of water

11.7.6 For standard #6 for the 200 ug/L standard, spike 80 uL of mix and 5 uL of surrogate to 40 mL of water.

11.7.7 For standard #7 for the 250 ug/L standard, spike 100 uL of mix and 6 uL of surrogate to 40 mL of water.

11.7.8 CCV standard, using the primary calibration standards from above, add 20 uL of mix to 40 mL of water

11.8 Secondary source stock standards shall be used to verify the initial calibration curve (ICV), and LCS and MS with duplicates. These standards shall be purchased from a different vendor, unless the primary vendor can supply different lot numbers that are verfied to be from a different source. This standard shall be at a concentration near the mid-level calibration standard.

11.8.1 Prepare secondary-source standards as noted above

11.8.2 Procedure for making MS solution:

11.8.2.1 Dilute 50 uL of 2000 ug/mL VOA Matrix Spike solution to 1 mL to give a final concentration of 100 ug/mL.

11.9 Preparation of MS (50 ug/L) sample using secondary source stock standards: matrix spiking standards should be prepared from volatile organic compounds that are representative of the compounds being investigated The matrix spiking solution should contain compounds that are expected to be found in the types of samples to be analyzed. In the absence of project related information, a representative subset of the target analytes may be used The following are the recommended subset for the matrix spike compounds:

1,1-dichloroethene
trichloroethene
chlorobenzene
toluene
benzene

12.0 Sample Preservation, Containers, Handling, and Storage

12.1 Samples for analysis using this SOP must be stored tightly sealed in VOA vials with Teflon-lined silicone septum seals in which the original sample was collected. All samples must be stored in the refrigerator at 4° ±2° C and be segregated from other samples Samples containing suspected high levels of fuels or other volatiles should be further segregated and stored separately from the other volatiles samples

12.2 Samples for volatiles analysis must be analyzed within 14 days of sample collection for preserved water samples and soils They must be analyzed within 7 days for unpreserved water samples.

13.0 Quality Control

13.1 A preparation batch of samples is defined as a group of up to twenty field samples of similar matrix type that have been prepared at the same time or time sequence with the same lots of reagents for the same analysis. In addition to the twenty samples, each preparatory batch will contain at a minimum, a method blank, a laboratory control sample, a matrix spike, a matrix spike duplicate and a matrix duplicate. An analytical, or instrumental batch is defined as samples that are analyzed together within the same time period or in continuous sequential time periods.

13.2 Perform BFB tune every 12 hours. Tuning acceptance criteria are presented in Section 16.1. The computer software will evaluate the tune information. The analyst should be aware of the process used. The criteria specified in Section 16.1 would typically be derived using three scans; one at the apex, and one on both sides of the apex

13.3 Run an initial calibration curve, using the primary source standards, each time major instrument maintenance occurs, or if either SPCC or CCC in the CCV do not meet acceptance criteria. Acceptance criteria are presented in Section 16.4 – 16.7.

13.4 Run an ICV sample, using secondary source standards, following the initial calibration curve. If SPCC or CCC in the ICV do not meet acceptance criteria, rerun the calibration curve. Acceptance criteria are presented in Section 16.4

13.5 Run a CCV sample, using primary standards, to determine if the instrument is still within calibration Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift. The SPCC and CCC in the CCV must meet acceptance criteria presented in Section 16.6.

13.6 A method blank must be prepared with each batch of samples The method blank should be prepared from either organic-free water or sand The method blanks are to be related to each 12-hour sequence of samples injected. Therefore, any given batch of samples may contain more than one method blank if that batch of samples is analyzed over any period of time that exceeds 12 hours. Acceptance criteria for these blanks are listed in Section 16.8 Instrument blanks may be injected at any time in the sequence to verify absence of contamination.

13.7 A laboratory control sample must be prepared and analyzed with each batch of samples. The LCS would be prepared using the matrix spiking standard Control charts will be maintained for the LCS for all target analytes and surrogate spikes Surrogate spike recoveries from the method blanks will also be control charted along with the LCS surrogate values. Acceptance criteria are presented in Section 16.9.

13.8 The use of the matrix spike and matrix spike duplicate will depend in part on what role the laboratory is playing. Acceptance criteria are presented in Section 16.10 – 16.11 for percent recovery and RPD.

13 8 1 When the laboratory serves the role as the primary laboratory, then site-specific documents should be consulted. The sample to be used for the MS/MSD may be specified in the field. This previously designated sample would then be spiked with the site-specific target analytes at a concentration equivalent to the site action level or lower. The MS/MSD would be prepared using the matrix spiking standard.

13 8.2 If this information was not specified or unknown, then the laboratory would choose a representative sample from each batch of samples analyzed

13 8 3 If samples from multiple sites were to be analyzed in the same batch, then multiple sets of MS/MSDs may be required.

13.8.4 When the laboratory serves the role as the QA laboratory, the above scenario may not be practical to implement

13.8.5 If the site-specific requirements are unknown and samples from multiple sites are analyzed in the same batch, then the laboratory should select a single sample for spiking from one of the projects Each batch of samples would then contain at least one MS/MSD pair

13 9 Surrogate recoveries are calculated for each LCS, method blank, QC sample, and field sample analyzed Acceptance criteria are listed in Section 16.14

13.10 The relative retention times (RRTs) of identified compounds need to be checked for each identified compound in samples, and compared to standard RRT Acceptance criteria are presented in Section 16.15

13.11 Ion abundances for target compounds and any tentatively identified compounds need to meet specific requirements Acceptance criteria for ion abundances are presented in Section 16 16.

13.12 Internal standard area counts for standards and samples must meet specifications as described in Section 16 17.

13.13 Data is checked to ascertain if it conforms to accepted practices. All sample analytical results used for final data reporting must be between the low standard and the high standard. Results that fall below the low standard or above the high standard are to be reported as estimated values. Corrective actions are described in Section 16.18.

13 14 MDLs are determined in either reagent water or organic-free sand / soil and verified annually. (Project-specific requirements may require that the MDL study be performed in the site-specific matrix.)

13.15 The analyst must demonstrate proficiency in performing the analysis. Method proficiency must be re-demonstrated anytime a major method modification is made, a major software revision is added, or a major instrument modification is made. Demonstration of method proficiency may also be required after major instrument maintenance. This is decided on a case-by-case basis through discussions with the Laboratory Technical Manager, and Quality Assurance Manager

14.0 Procedure

14 1 Before analysis can begin, a BFB tune of the GC/MS must be done. The system must be hardware-tuned with BFB to meet the criteria in Section 16.2. Since BFB is in the surrogate mix, a blank can be run and the BFB analyzed from that blank. The mass-spectra must meet acceptance criteria in 16.1 before analyses can begin All standards shall be analyzed within the same 12-hour tune shift.

14 2 Initial calibration, as required in section 16.1 through 16.7.

14.2.1 Prepare a calibration curve; refer to section 11.7 for preparing calibration standards. Other calibration levels could be chosen between these levels based on the project specific requirements. Section 16 1 4 provides the calibration curve requirements.

14.2.1.1 Fill a gas-tight syringe (GTS) with 5mL of water.

14.2.1.2 Open the Luer tip and inject the various standards listed in section 11 7 into the GTS.

14.2.1.3 Inject GTS into purge tube corresponding to the appropriate standard on the autosampler.

14.2.1.4 Close valve on autosampler and analyze standards.

14.2 2 Prepare a 50ug/L ICV sample

14.2.2.1 Fill a gas-tight syringe (GTS) with 5mL of water.

14.2.2.2 Open the Luer tip and inject the secondary standard into the GTS

14.2.2.3 Inject GTS into purge tube corresponding to the appropriate standard on the autosampler.

14.2.2 4 Close valve on autosampler and analyze standards.

Note: Injections should be performed from the lowest to the highest standards with a blank injected after the highest standard and followed by the secondary source standard to verify the initial calibration curve.

14.2 3 Tabulate the area response of the characteristic ions against each compound's concentration and each internal standard concentration. Then calculate the response factor (RF) for the quantifying ion of each compound relative to the appropriate internal standard according to the calculation provided in Section 15.1 The internal standards used should permit most of the compounds of interest in a chromatogram to have retention times of 0.80 to 1.20, relative to one of the internal standards. The average RF must be calculated and recorded for each compound. Check performance criteria found in section 16.1 3.

14.2.4 The continuing calibration verification (CCV) is prepared using the secondary source standards. This should be prepared near the mid-point of the calibration standard The results from the CCV should meet the verification requirements that are the same as the requirements for the SPCC and CCC noted in section 16 3.

14.2 5 A system performance check is to be made before this calibration curve is used. Five SPCC compounds are checked for a minimum average response factor The minimum relative response factor for volatile SPCCs are as follows:

Chloromethane	0 10
1,1-Dichloroethane	0 10
Bromoform	0 10
Chlorobenzene	0 30
1,1,2,2-Tetrachloroethane	0 30

These compounds are typically used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

14.2.5.1 Chloromethane – This compound is the most likely compound to be lost if the purge flow is too fast.

14.2.5.2 Bromoform – This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

14.2.5.3 Tetrachloroethane and 1,1-dichloroethane – These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

14.2.6 Adjust the purge gas (helium) flow rate to 25-40mL/min on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response.

14.2.7 The Continuing Calibration Verification (CCV) check compounds are run to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may indicate a system leak or reactive sites on the column. The %RSD for each individual CCV must be less than 25%. The CCC are:

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

14.2.7.1 Preparation of a CCV sample uses secondary source standards.

14.2.7.2 Fill GTS with 5mL of water.

14.2.7.3 Open Luer tip and inject 5uL of 50ppm IS, 2.5uL of the 100ppm multi-mix solution, 2.5uL of the 100ppm surrogate solution, 2.5uL of the 100ppm gases solution, and 2.5uL of the 200ppm acrolein solution into the GTS.

14.2.7.4 Inject GTS into corresponding purge tube on autosampler.

14.2.7.5 Close valve and analyze samples.

14.3 Sample preparation (water)

14.3.1 Withdraw 5mL of sample into a GTS.

14.3.2 Open Luer tip and inject 1 uL of 250 ug/mL of Internal Standard and Surrogate Mix into the GTS.

14.3.3 Inject GTS into corresponding purge tube on autosampler.

14.3.4 Close valve and analyze sample

14.3.4.1 MS/MSD samples are prepared by withdrawing 5mL of sample into a GTS

14.3.4.2 Open Luer tip and inject 1 uL of 250 ug/mL of Internal Standard Surrogate Mix

14.3.4.3 Inject GTS into corresponding purge tube on autosampler

14.4 Soil sample preparation (Low concentration)

14.4.1 Bring samples to room temperature; low concentration soils require a heated purge to be used.

14.4.2 Weigh 5g of sample into a purge tube.

14.4.3 Attach purge tube to autosampler

14.4.4 Fill GTS with 5mL of water.

14.4.5 Open Luer tip and inject 1 uL of 250 ug/mL of Internal Standard Surrogate Mix.

14.4.6 Inject GTS into corresponding purge tube on autosampler.

14.4.7 Close valve and analyze

14.4.7.1 MS/MSD samples are prepared following step 14.4.1 through 14.4.3.

14.4.7.2 Open Luer tip and inject 1 uL of 250 ug/mL of Internal Standard Surrogate Mix, and 2.5uL of 100ppm MS solution into the GTS

14.4.7.3 Inject GTS into corresponding purge tube on autosampler.

14.4.7.4 Close valve and analyze

14.4.7.5 Alternatively, autosampler is used to inject IS + Surrogate automatically.

14.5 Soil sample preparation (Medium concentration). If <5 medium contaminated soils require methanol extraction, continue with the following steps. If ≥5 samples need methanol extraction, follow step 14.6.

14.5.1 Bring samples to room temperature

14.5.2 Weigh out 5 grams (to the nearest 0.01g) of the sample into a 40mL VOA vial

14.5.3 Add 5mL of methanol into the vial.

14.5.4 Seal with a cap and septum, and then gently swirl the vial.

14.5.5 Allow the soil to settle

14.5.6 Add 5mL of water to another vial.

14.5.7 Remove the appropriate volume of extract from the first vial using a gastight syringe. The dilution factor equals the total volume of water and extract, divided by the volume of extract. Example, 50uL of MeOH extract in 5mL sample = 5050uL/50uL = 101 dilution factor.

14.5.8 Transfer extract to the second vial.

14.5.9 Prepare MS/MSD samples.

14.5.9.1 Add 5mL of water to additional vials.

14.5.9.2 Transfer the same volume of extract to the vials.

14.5.9.3 Spike samples with MS solution to a final concentration of 50 ug/mL.

14.5.10 Prepare a methanol method blank. This blank is exclusively for extracted samples.

14.5.10.1 Add 5mL of water to a new vial

14.5.10.2 Transfer the same volume as in step 14.5.6 of clean methanol to a new vial.

14.5.10.3 The concentration of any target analyte must be below the reporting limit. If not, contamination has occurred. Follow steps in section 16.8 for corrective actions

14.6 Soil sample preparation (Medium concentration) If <5 medium contaminated soils require methanol extraction, follow step 14.5. If ≥5 samples need methanol extraction, continue with the following steps

14.6.1 Weigh out 5 grams (to the nearest 0.01 g) of the sample into a 40mL VOA vial.

14.6.2 Add 10 mL of methanol into the vial

14.6.3 Seal with a cap and septum, and then gently swirl the vial

14.6.4 Allow the soil to settle

14.6.5 Add 5mL of water to another vial

14.6.6 Remove the appropriate volume of extract from the first vial using a gastight syringe The dilution factor equals the total volume of water and extract, divided by the volume of extract Example, 50uL of MeOH extract in 5mL sample = 5050uL/50uL = 101 dilution factor

14.6.7 Transfer the extract to the second vial.

14.6.8 Prepare MS/MSD samples.

14.6.8.1 Add 5mL of water to additional vials.

14.6.8.2 Transfer the same volume of extract to each vial.

14.6.8.3 Spike samples with MS solution to a final concentration of 50 ug/mL.

14.6.9 Prepare an initial calibration curve, ICV, MB, LCS, and CCV for soils. Incorporate the same volume of methanol used in step 14.6.6 into each calibration and QC sample. If the volume of

methanol varies with soil sample, incorporate the largest volume into each sample. The QC samples in this preparation batch must meet all QC acceptance criteria.

14.7 Preparation of a LCS sample using secondary source standards.

14.7.1 Fill GTS with 5mL of water.

14.7.2 Open Luer tip and inject 5uL of 50ppm IS, 2.5uL of the 100ppm multi-mix solution, 2.5uL of the 100ppm surrogate solution, 2.5uL of the 100ppm gases solution, and 2.5uL of the 200ppm acrolein solution into the GTS

14.7.3 Inject GTS into corresponding purge tube on autosampler.

14.7.4 Close valve and analyze samples.

14.8 Preparation of method blank sample.

14.8.1 Fill GTS with 5mL of water

14.8.2 Open Luer tip and inject 5uL of 50ppm IS, and 2.5uL of the 100ppm surrogate solution into the GTS

14.8.3 Inject GTS into corresponding purge tube on autosampler.

14.8.4 Close valve and analyze samples

14.9 Qualitative Identification

The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. All hits should be visually compared to the reference spectrum for confirmation The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met.

14.9.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion

14.9.2 The RRT of the sample component is within ±0.06 RRT units of the RRT of the standard component

14.9.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

14.9.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is

achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

14.9.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criterion can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

14.9.6 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. When serving the role of QA (or referee) laboratory, tentatively identified compounds (TICs) shall always be reported. Guidelines for making tentative identification are:

14.9.6.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

14.9.6.2 The relative intensities of the major ions should agree within ± 30% to be consistent with target compound list identification. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%).

14.9.6.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

14.9.6.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

14.9.6.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

14.9.6.6 Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

14.10 Quantitative analysis

14.10.1 When a compound has been identified, the quantitation of that compound is based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation is accomplished using the internal standard technique, as described in sections 15.5 and 15.6. The internal standard used must be the one nearest the retention time of that of a given analyte.

14.10.2 Sediment/soil samples are reported on a dry weight basis, while sludges and wastes are reported on a wet weight basis. The percent dry weight of the sample (see section 15.7) must be reported along with the data in either instance.

15.0 Calculations

15.1 Response Factor (RF)

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s	=	Peak area (or height) of the analyte or surrogate.
A_{is}	=	Peak area (or height) of the internal standard.
C_s	=	Concentration of the analyte or surrogate, in µg/L.
C_{is}	=	Concentration of the internal standard, in µg/L.

15.2 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{SD}{\overline{RF}}$$

where:

RSD	=	Relative standard deviation.
$\overline{RF}$	=	Mean of 5 initial RFs for a compound
SD	=	Standard deviation of average RFs for a compound.

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RF}\right)^2}{n-1}}$$

15.3 Percent Difference

$$\%\ Difference = \frac{\left(\overline{RF} - RF_v\right)}{\overline{RF}} \times 100$$

where:

RF_v	=	Response factor from verified standard
$\overline{RF}$	=	Mean response factor from the initial calibration.

15.4 Percent Drift

$$\%\ Drift = \frac{\text{Calculated Conc.} - \text{Theoretical Conc.}}{\text{Theoretical Conc.}} \times 100\%$$

15.5 Concentration of Target Analytes in Water and Water-Miscible Waste

$$Concentration = \frac{A_x \times I_s \times D}{A_{is} \times \overline{RF} \times V_s}$$

where:

A_x	=	Area of the peak for the analyte in the sample
A_{is}	=	Area of the peak for the internal standard.
I_s	=	Mass (amount) of the internal standard in the concentrated sample extract (ng). This is not just the mass injected into the instrument, but the total mass of internal standard in the concentrated extract
D	=	Dilution factor, if the sample or extract were diluted prior to analysis. If no dilutions were made, D = 1. The dilution factor is always dimensionless.
$\overline{RF}$	=	Mean response factor from the initial calibration.
V_s	=	Volume of the aqueous sample extracted or purged (mL). If liter units are used for this term, multiple 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.

15.6 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste

$$\text{Concentration} = \frac{A_x \times I_s \times V_t}{A_{is} \times \overline{RF} \times V_i \times W_s}$$

where:

A_x, I_s, A_{is}, $\overline{RF}$ are the same as in water and water-miscible waste above.

V_t	=	Volume of total extract (µL) (use 10,000 µL or a factor of this when dilutions are made).
V_i	=	Volume of extract added (µL) for purging.
W_s	=	Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/Kg.

15.7 Percent dry weight

$$\%\ \text{Dry Weight} = \frac{\text{grams of dry sample}}{\text{grams of sample}} \times 100$$

15.8 Calculation of Recovery

$$\%\text{Recovery} = \frac{C_m}{C_s} \times 100$$

where:

C_m = Measured concentration of the surrogate
C_s = Nominal (or theoretical) Concentration of the surrogate

For MS/MSD and LCS analyses:

$$\%\text{Recovery} = \frac{C_s - C_u}{C_n} \times 100$$

where:
C_s = Measured concentration of the spiked sample aliquot
C_u = Measured concentration of the unspiked sample aliquot (use 0 for LCS analyses)
C_n = Nominal (or theoretical) concentration of the spiked sample aliquot

15.9 **Calculation of Precision**

$$\%\text{RPD} = \frac{(C_1 - C_2)}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where:
C_1 = Measured concentration of the first sample
C_2 = Measured concentration of the second sample

15.10 **Linear regression**

15.10.1 Step 1: minimize the sum of the squares

$$S_{xx} = \sum \left(x_i - \bar{x}\right)^2$$

where:
S_{xx} = sum of squares for concentration of standard solution
X_i = individual data points for concentration
$\overline{X}$ = average value for concentrations, see 10.3

$$S_{yy} = \sum \left(y_i - \bar{y}\right)^2$$

where:
S_{yy} = sum of squares for area of a peak
Y_i = individual data points for area of peak
$\overline{Y}$ = average value for area of peaks, see 10.3

$$S_{xy} = \sum \left(X_i - \overline{X}\right)\left(Y_i - \overline{Y}\right)$$

where:
S_{xy} = sum of squares for concentration of standard solution
X_i = individual data points for concentration
$\overline{X}$ = average value for concentrations, see 10.3
Y_i = individual data points for area of peak
$\overline{Y}$ = average value for area of peak, see 10.3

15.10.2 Step 2: calculate the slope and intercept

$$m = \frac{S_{xx}}{S_{yy}}$$

where:
S_{xx} = sum of squares for concentration of standard solution
S_{yy} = sum of squares for area of a peak
m = slope of the line

$$b = \overline{Y} - m\overline{X}$$

where:
$\overline{Y}$ = average value for area of peak
$\overline{X}$ = average value for concentration
m = slope of the line
b = intercept of line

15.10.3 Step 3, calculate the least-squares line

$$Y = mX + b$$

where:
Y = calculated peak areas
m = least-squares slope
b = least-squares intercept
X = concentrations

15.11 **Percent Drift**

$$\% \text{ Drift} = \frac{\text{Calculated concentration - Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

16.0 Data Assessment and Corrective Actions

Site-specific requirements must be checked and used, if known, for data review. The criteria presented in this section should be used as a default list in the absence of site-specific requirements

16.1 Ensure that the BFB tune was run at the beginning of each 12-hour sequence for each batch of samples analyzed. The acceptance criteria are listed below:

BFB Key Ions and Ion Abundance 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	< 2.0% if mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174

177	5.0-9.0% of mass 176

16.1.1 If the BFB acceptance criteria are not met, perform any or all of the following corrective actions:

- Re-inject BFB
- Retune with PFTBA, and then re-inject BFB.
- Clean MS source, retune with PFTBA, and re-inject BFB

16.1.2 If the tuning criteria still cannot be met after performing the above, have the mass spectrometer serviced by manufacturer representative

16.1.3 Using the RFs from the initial calibration, calculate and record the percent relative standard deviation (%RSD) for all calibration compounds using equation 15.2. The %RSD should be less than 15% for each compound, then linearity is confirmed and the relative response factor is assumed constant over the calibration range. The average relative response factor may be used for quantitation

16.1.4 If a %RSD >15%, but ≤30%, plot and/or visual inspect the calibration data. Plotting this data is a useful diagnostic tool. Analytical problems are discovered, including errors in standard preparation, the presence of active sites in the chromatographic system, and analytes that exhibit poor chromatographic behavior. If the RSD of ANY compound exceeds 15% (never to exceed 30%)during the initial calibration, then one of the following options must be applied to the GC/MS initial calibration in this situation, or a new initial calibration must be performed.

16.1.4.1 Adjust the instrument and/or perform instrument maintenance until the RSD of the calibration meets the 15% QC limit. This option would apply in those instances where a linear instrument response is expected

16.1.4.2 Narrow the calibration range until the response is linear. If the low standard is below the estimated quantitation limit (i.e., for the poor purgers in a commercially available prepared standard mix), then this standard may be dropped. Recalculate the RSD without this standard to see if the RSD meets the QC limit. It would be recommended that a new standard be prepared at a concentration between the existing fourth and fifth calibration standards, analyzed, and a new RSD calculated with all five points. If this is not practical, then the use of four points for the initial calibration curve for the poor purgers would be acceptable.

16.1.4.3 Use a linear calibration that does not pass through the origin. This would be achieved by performing a linear regression of the instrument response versus the concentration of the standards. Follow the calculation in section 15.10 to determine this line. The line should not be forced through the origin and the origin should not be included as a sixth calibration point. In order to be used for quantitative purposes, the correlation coefficient must be greater than or equal to 0.99.

16.1.4.4 Use a calibration curve or non-linear calibration model. This option should be used only after exhausting the other three options, or in situations where the analyst knows that the instrument response does not follow a linear model over the width of the working range

16.2 If a %RSD >30 percent is measured for any target analytes during calibration, then corrective action to eliminate the problem (system leak and/or column reactive sites) is required before reattempting calibration.

16.3 An initial calibration verification (ICV) sample must be analyzed after performing an initial calibration. If either SPCC or CCC criteria are not met, reanalyze the ICV or prepare a new calibration curve as necessary.

16.3.1 SPCCs must meet acceptance criteria for RF in section 14.2.5.

16.3.2 CCCs must meet the following criteria to validate the initial calibration. The percent difference criterion must be met when calibration is based on an average response factor. The percent drift criterion must be met when calibration is based on a regression fit model.

- The percent difference or percent drift for all target analytes in the ICV sample should be ±20%, if CCCs are not in the reported list.
- The percent difference or percent drift for CCC in the ICV sample must be ±20%.

16.4 In a CCV, if a %RSD is greater than 20 percent for any target analyte listed in section 14.2.7, then corrective action to eliminate a system leak and/or column reactive sites is required before reattempting calibration.

16.5 System Performance Check Compounds (SPCCs): A system performance check must be made during every 12-hour shift. This check is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum RF for volatile SPCCs is listed in Section 14.2.5.

16.6 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs listed in Section 14.2.7 are used to check the validity of the initial calibration. The percent difference criterion should be applied to all method target analytes, not just the CCCs. The acceptance criterion is <20% drift for the target analytes.

16.7 If this criterion is exceeded, corrective action is necessary. If the source of the problem cannot be determined after corrective action has been taken, a new five-point calibration must be generated. This criterion must be met before sample analysis begins and/or reanalysis of all samples up to the last acceptable CCV standard.

16.8 Assess the method blanks. The analyst shall confirm that this blank was analyzed at the required frequency. The method blank should not exhibit any contamination of any analyte above the MDL for any of the method target analytes. Corrective action should be performed any time method target analytes are detected above the MDL to reduce and control contamination. Corrective action will be required if site-specific target analytes are detected at greater than 5% of the regulatory limit for that analyte or if the concentration in the blank is greater than 5% of that in the sample. The first step of corrective action is to assess the effect on the samples. Corrective action would include reanalysis of field and QC samples in the batch if some or all of the samples also contained levels of target analytes that exceeded the above criterion. If none of the field samples had values above the stated criteria, then reanalysis may not be necessary. The source of contamination should still be investigated and reduced/eliminated. Any time contamination is noted in the method blank, the situation and impact on the data should be discussed in the case narrative.

16.9 Assess that LCSs were prepared at the required frequency. Plot the target compounds on appropriate control charts. If all target compounds are not within control limits, the affected samples are to be reanalyzed for verification. If it is still out of control limits, then all field and QC samples in the batch must be reanalyzed. Even though control charts must be maintained for the LCSs, the acceptance criteria derived from these charts may not be appropriate if the charted ranges are too wide. The range is 70–130%. The RPD of interbatch LCSs should fall within the control limits determined from the precision control charts. However, if the RPD is outside these control limits, the batch will not be

rejected, as long as the LCS recovery is acceptable. This precision information should be evaluated to see systematic problems could be identified If problems are suspected, the method should be fully evaluated.

16.10 Assess that matrix spike/matrix spike duplicates were analyzed at required frequency. Acceptance criteria are that all % Recovery and/or RPD results meet project-established goals. If no project goals are specified, then results must be within the indicated control limits on the appropriate LCS control charts. If these conditions are not met, perform the following corrective actions as appropriate. The analyst shall also verify that the samples were spiked at the appropriate level. The order of preference for spiking levels is as follows.

16.10.1 If the target analyte concentrations are known, spike to increase the background concentration by a factor of approximately two

16.10.2 If an action level exists, spike at this level

16.10.3 If neither of the first two conditions apply, spike at a level that corresponds between the low and mid-level calibration standards

16.11 If both LCS and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be reanalyzed. If the MS/MSD is unacceptable, but the LCS is acceptable, then a potential matrix effect has been identified. Reanalyze to verify matrix effect. If a matrix effect is still suspected, then the project manager must be contacted to discuss further alternatives and the potential impact on the project. Reported data should be flagged. Reasonable attempts must be made to address matrix interference

16.12 Assess that matrix duplicates were analyzed at required frequency. Acceptance criteria are that all RPD results meet project-established goals. If no project goals are specified, then results must be within the indicated control limits on the appropriate LCS precision control charts. If these conditions are not met, perform the following corrective actions as appropriate.

16.13 Reanalyze the sample to verify a matrix effect. If the duplicate precision is still unacceptable, and LCS precision is acceptable, then a potential matrix effect has been identified. The project manager must be contacted to discuss further alternatives and the potential impact on the project.

16.14 Check the surrogate calculations for correctness for all samples, blanks, LCS, MS, MSD, and MD. Check that the surrogate recoveries are properly control-charted for the LCS and method blank(s). The following acceptance criteria apply to surrogate recoveries.

16.14.1 The surrogate recoveries for all field and QC samples must be within control limits on control charts. If the LCS and/or method blank recoveries are outside limits, reanalysis must be performed for verification. If still out, corrective action is necessary.

16.14.2 Sample surrogate recoveries are compared to the LCS surrogate control limits. If sample surrogate recoveries exceed these limits, the sample must be reanalyzed. If the results are still outside the limits, then the project manager is to be contacted to discuss further alternatives and the impact on the project.

16.14.3 Even though control charts must be maintained for the LCS and blanks, the acceptance criteria derived from these charts may not be appropriate if the charted ranges are too wide. A maximum default range should be used. This range should also be compared to the guidance limits as stated in the method. It is recommended that the maximum default range be set to 70 to 130%. A typical default range should be closer to 75 to 125%. Recovery ranges for soils will be wider than for waters.

16.15 The relative retention times must be checked for all identified compounds in both standards and samples. The internal standard absolute retention times must also be checked for all analyses. Acceptance criteria are as follows:

16.15.1 The relative retention times of each compound in each calibration run should agree within ±0.06 relative retention time (RRT) units. Late-eluting compounds usually have much better agreement.

16.15.2 If the retention time for any internal standard changes by more than 30 seconds from the last daily calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

16.16 The analyst must verify that ion abundances meet specific criteria for the various analyses. The following acceptance criteria shall be checked for all appropriate samples.

16.16.1 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

16.16.2 The relative intensities of ions must agree within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample abundance must be between 20 and 80 percent.)

16.16.2.1 Molecular ions present in the reference spectrum should be present in the sample spectrum.

16.16.2.2 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

16.16.2.3 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

16.16.2.4 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. When serving the role as QA (or referee) laboratory, tentatively identified compounds (TICs) are always reported. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

16.16.2.5 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

16.16.2.6 The relative intensities of the major ions should agree within ± 30%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%.)

16.16.2.7 Molecular ions present in the reference spectrum should be present in the sample spectrum

16.16.2.8 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

16.16.2.9 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

16.17 The analyst will check the internal standard area counts for all calibration standards, QC samples, and samples for quantitation. If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from the last daily calibration standard check, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. In the event the internal standard area counts fail this criterion, the following corrective actions should be considered.

16.17.1 Check to be sure there are no errors in internal standards preparation or addition. Also check instrument performance. Recalculate the data and/or reanalyze the sample if any of the above checks reveal a problem.

16.18 The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot.

17.0 Waste Disposal and Pollution Prevention

17.1 This procedure generates some organic solvent wastes from unused standards and non-aqueous samples and solid waste from solid samples. All waste disposal procedures must comply with federal and local regulations. Refer to the Sample and Waste Disposal SOP A-2050.

17.2 Pollution prevention is any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. This procedure generates solvent waste and uses solvent based standards. The laboratory should minimize the amount of standards and solvents used whenever possible. Small volumes of standards are used to create calibration and QC standards. Concentrated standards should be ordered in quantities sufficient to be fully utilized by their expiration date, to eliminate unnecessary disposal of concentrated metals standards.

18.0 References

18.1 Method 8260A, Test Methods for the Analysis of Solid Waste, Third Edition, Update II, September 1994.

18.2 Method 8000B, Test Methods for the Analysis of Solid Waste, Physical/Chemical Methods, Update II, Third Edition.

18.3 EPA Method 624: 40 CFR Ch1 Pt 136 App. A

VOC MDL Study Aqueous 2007

Analysis Method: SW846-8260B

Prep Method: SW846-8260B **Instrument:** Varian GC/MS

Liquid Matrix			Replicate Number							Samples	Mean	Average	Deviation	MDL	Reporting Limit
Compound	Est. Limit (ug/L)	Spike Conc. (ug/L)	1	2	3	4	5	6	7	(N)	(ug/L)	%Rec	(s)	(ug/L)	(ug/L)
		Prep Date	2/22/2007	2/22/2007	2/23/2007	2/23/2007	3/1/2007	3/1/2007	3/2/2007						
		Analysis Date	2/22/2007	2/22/2007	2/23/2007	2/23/2007	3/1/2007	3/1/2007	3/2/2007						
Dichlorodifluoromethane**	5	20.00	18.598	21.649	15.057	22.909	22.956	25.884	26.202	7	21.89	109.5%	3.67	11.54	20.00
Chloroethane	5	20.00	21.510	24.790	23.270	28.080	22.000	23.490	26.000	7	24.16	120.8%	2.14	6.73	10.00
Vinyl chloride	5	20.00	19.270	19.290	20.320	19.970	21.120	19.980	20.590	7	20.08	100.4%	0.62	1.95	5.00
Bromomethane	5	20.00	15.300	14.450	21.300	22.010	21.850	18.910	22.260	7	19.44	97.2%	3.07	9.66	10.00
Chloromethane	5	20.00	17.120	18.450	21.180	21.130	19.050	19.430	20.170	7	19.50	97.5%	1.36	4.27	5.00
Trichlorofluoromethane	5	20.00	20.660	20.930	19.460	19.870	19.760	19.590	19.800	7	20.01	100.1%	0.52	1.63	5.00
Acrolein	100	100.00	100.890	103.920	89.870	91.250	83.670	86.570	93.380	7	92.79	92.8%	6.79	21.34	100.00
Iodomethane	5	20.00	19.000	20.590	19.440	19.800	17.840	17.810	19.580	7	19.15	95.8%	0.95	2.98	5.00
Acrylonitrile	100	100.00	151.150	156.800	132.050	135.020	135.670	145.470	146.090	7	143.18	143.2%	8.53	26.82	100.00
Acetone	10	40.00	45.100	45.750	40.320	40.650	40.760	40.900	43.150	7	42.38	105.9%	2.12	6.66	50.00
1,1-Dichloroethene	5	20.00	23.140	22.750	19.640	21.820	19.570	17.450	17.260	7	20.23	101.2%	2.23	7.00	100.00
Carbon Disulfide	5	20.00	21.550	22.220	18.420	18.210	18.830	18.560	18.750	7	19.51	97.5%	1.53	4.80	5.00
Methylene Chloride**	5	20.00	20.370	19.557	20.525	19.651	19.424	19.166	20.392	7	19.87	99.3%	0.51	1.59	5.00
trans-1,2-Dichloroethene	5	20.00	21.963	25.293	25.999	22.014	27.064	25.740	27.481	7	25.08	125.4%	2.08	6.52	10.00
Methyl-tert-Butylether (MTBE)**	5	20.00	21.832	21.384	22.500	22.293	22.624	21.988	23.183	7	22.26	111.3%	0.54	1.71	5.00
1,1-Dichloroethane	5	20.00	25.450	24.240	22.610	21.080	19.910	21.890	20.660	7	22.26	111.3%	1.85	5.80	10.00
Vinyl acetate	10	40.00	66.444	65.436	64.255	64.667	66.499	66.404	72.077	7	66.54	166.4%	2.41	7.57	10.00
Methyl Ethyl Ketone	10	40.00	50.720	49.850	42.570	42.930	43.660	44.450	48.960	7	46.16	115.4%	3.27	10.27	100.00
cis-1,2-Dichloroethene**	5	20.00	22.553	23.686	24.347	21.832	24.861	24.362	25.547	7	23.88	119.4%	1.21	3.79	5.00
2,2-dichloropropane**	5	50.00	28.830	34.285	34.820	28.071	35.926	35.257	38.592	7	33.68	67.4%	3.55	11.16	50.00
Chloroform	5	20.00	23.480	23.860	21.400	21.340	22.270	22.000	22.680	7	22.43	112.2%	0.90	2.83	50.00
Bromochloromethane	5	20.00	21.020	21.760	19.730	19.930	16.770	18.900	21.300	7	19.92	99.6%	1.58	4.97	5.00
1,1,1-Trichloroethane	5	20.00	22.110	23.590	20.100	20.190	20.650	19.970	20.570	7	21.03	105.1%	1.24	3.90	100.00
1,1-Dichloropropene	5	20.00	21.330	22.370	19.680	20.590	19.750	18.710	18.680	7	20.16	100.8%	1.26	3.96	5.00
Carbon Tetrachloride	5	20.00	21.390	22.690	19.740	19.670	21.490	20.700	20.690	7	20.91	104.6%	0.98	3.08	5.00
1,2-Dichloroethane	5	20.00	26.230	26.800	23.420	22.960	22.800	23.090	23.510	7	24.12	120.6%	1.54	4.85	5.00
Benzene	5	20.00	21.560	23.190	20.090	19.790	19.400	19.490	20.150	7	20.52	102.6%	1.28	4.01	5.00
Trichloroethene	5	20.00	20.290	21.720	20.890	20.120	19.490	19.370	19.540	7	20.20	101.0%	0.80	2.50	5.00
1,2-Dichloropropane**	5	20.00	20.359	21.997	22.303	21.429	22.446	22.794	23.337	7	22.10	110.5%	0.90	2.83	5.00
Dibromomethane**	5	20.00	21.691	22.418	22.430	22.471	22.797	22.985	22.834	7	22.52	112.6%	0.40	1.25	5.00
Bromodichloromethane	5	20.00	20.810	22.440	21.040	20.480	21.330	20.510	21.010	7	21.09	105.4%	0.62	1.94	5.00
cis-1,3-Dichloropropene	5	20.00	20.580	21.630	20.790	20.530	19.430	19.620	20.440	7	20.43	102.2%	0.68	2.14	5.00
MIBK**	10	40.00	42.584	40.143	40.539	42.902	42.816	43.231	43.226	7	42.21	105.5%	1.20	3.78	5.00
Toluene	5	20.00	18.980	20.280	19.100	18.190	18.230	17.980	18.470	7	18.75	93.7%	0.73	2.31	5.00
trans-1,3-Dichloropropene	5	20.00	21.150	21.140	21.170	20.940	18.600	19.330	21.080	7	20.49	102.4%	0.98	3.10	5.00
1,1,2-Trichloroethane	5	20.00	22.830	23.250	21.020	20.910	21.180	20.910	21.440	7	21.65	108.2%	0.90	2.84	10.00
Tetrachloroethene	5	20.00	20.700	21.030	19.480	19.640	21.170	22.110	19.970	7	20.59	102.9%	0.88	2.75	5.00
2-Hexanone	10	40.00	46.090	44.850	42.480	43.450	45.910	48.250	49.700	7	45.82	114.5%	2.35	7.40	10.00
Dibromochloromethane	5	20.00	24.590	24.750	23.640	22.610	23.740	24.650	24.670	7	24.09	120.5%	0.74	2.33	5.00
1,2-Dibromoethane	5	20.00	22.890	22.730	20.700	20.640	21.200	21.560	22.280	7	21.71	108.6%	0.86	2.71	50.00
Chlorobenzene	5	20.00	19.150	20.480	20.450	20.310	19.770	19.510	19.250	7	19.85	99.2%	0.53	1.65	5.00
1,1,1,2-Tetrachloroethane	5	20.00	24.790	23.870	21.020	21.360	21.140	20.750	21.500	7	22.06	110.3%	1.47	4.63	5.00
Ethylbenzene	5	20.00	19.550	20.820	20.030	20.340	19.970	19.300	19.080	7	19.87	99.4%	0.56	1.77	5.00
m,p-Xylenes	10	40.00	39.620	41.370	40.570	40.680	39.730	38.950	37.880	7	39.83	99.6%	1.09	3.42	5.00
o-Xylene	5	20.00	19.800	20.890	22.520	22.240	19.920	19.780	19.090	7	20.61	103.0%	1.23	3.85	5.00
Styrene	5	20.00	19.020	19.660	19.350	19.780	19.100	18.390	18.070	7	19.05	95.3%	0.58	1.84	20.00
Bromoform	5	20.00	20.270	21.060	20.500	20.090	20.120	20.640	20.270	7	20.42	102.1%	0.32	1.00	5.00
Isopropyl Benzene	5	20.00	19.000	20.050	19.400	19.440	19.230	19.240	18.400	7	19.25	96.3%	0.46	1.45	5.00
1,1,2,2-Tetrachloroethane	5	20.00	21.600	22.320	20.950	21.960	21.140	22.150	21.900	7	21.72	108.6%	0.47	1.49	5.00

VOC MDL Study Aqueous 2007

Analysis Method: SW846-8260B

Prep Method: SW846-8260B **Instrument:** Varian GC/MS

Liquid Matrix			Replicate Number							Samples	Mean	Average	Deviation	MDL	Reporting Limit
Compound	Est. Limit (ug/L)	Spike Conc. (ug/L)	1	2	3	4	5	6	7	(N)	(ug/L)	%Rec	(s)	(ug/L)	(ug/L)
		Prep Date	2/22/2007	2/22/2007	2/23/2007	2/23/2007	3/1/2007	3/1/2007	3/2/2007						
		Analysis Date	2/22/2007	2/22/2007	2/23/2007	2/23/2007	3/1/2007	3/1/2007	3/2/2007						
Bromobenzene	5	20.00	23.860	24.060	20.380	20.690	20.890	20.600	21.450	7	21.70	108.5%	1.46	4.59	5.00
1,2,3-Trichloropropane**	5	20.00	18.953	19.000	20.040	19.973	20.128	18.617	18.697	7	19.34	96.7%	0.62	1.96	5.00
n-Propylbenzene	5	20.00	23.550	23.330	20.560	20.880	20.140	20.460	20.880	7	21.40	107.0%	1.31	4.13	10.00
2-Chlorotoluene	5	20.00	24.510	23.950	20.820	21.010	20.950	20.980	21.560	7	21.97	109.8%	1.45	4.57	5.00
1,3,5-trimethylbenzene	5	20.00	23.690	23.680	20.600	20.480	20.190	20.480	21.320	7	21.49	107.5%	1.42	4.47	5.00
4-Chlorotoluene	5	20.00	22.850	23.300	20.350	21.120	19.390	20.330	20.070	7	21.06	105.3%	1.36	4.29	5.00
tert-Butylbenzene**	5	20.00	18.647	22.880	23.768	19.098	22.901	23.098	21.728	7	21.73	108.7%	1.90	5.96	10.00
1,2,4-Trimethylbenzene	5	20.00	24.000	23.500	20.790	21.250	20.750	20.970	21.000	7	21.75	108.8%	1.28	4.02	5.00
sec-Butylbenzene	5	20.00	22.950	23.320	19.910	20.800	19.970	20.220	20.030	7	21.03	105.1%	1.36	4.29	5.00
p-Isopropyltoluene	5	20.00	22.850	23.300	20.350	21.120	19.390	20.330	20.070	7	21.06	105.3%	1.36	4.29	5.00
1,4-Dichlorobenzene	5	20.00	19.410	20.530	22.000	20.700	18.940	19.530	19.110	7	20.03	100.2%	1.02	3.20	5.00
1,3-Dichlorobenzene	5	20.00	19.410	20.530	21.600	21.140	19.950	20.310	20.100	7	20.43	102.2%	0.68	2.15	5.00
n-Butylbenzene	5	20.00	21.990	22.610	20.030	21.030	18.540	19.850	19.790	7	20.55	102.7%	1.31	4.10	5.00
1,2-Dichlorobenzene	5	20.00	19.740	21.510	21.810	21.570	19.850	20.570	19.650	7	20.67	103.4%	0.88	2.76	5.00
1,2-Dibromo-3-chloropropane	5	20.00	17.300	22.060	17.220	22.560	17.270	22.490	17.970	7	19.55	97.8%	2.46	7.72	5.00
1,2,4-Trichlorobenzene	5	20.00	19.719	23.679	25.756	15.549	22.150	14.602	21.198	7	20.38	101.9%	3.79	11.92	20.00
Hexachlorobutadiene	5	20.00	24.491	28.813	23.078	23.729	27.434	21.365	25.113	7	24.86	124.3%	2.36	7.43	10.00
Naphthalene	5	20.00	23.555	28.410	31.626	18.219	26.013	17.231	25.381	7	24.35	121.7%	4.81	15.13	20.00
1,2,3-Trichlorobenzene	5	20.00	20.860	24.390	16.120	23.150	13.220	20.100	15.690	7	19.08	95.4%	3.85	12.09	20.00

** Analyzed and Extracted on 5/2/07

VOC MDL Study Solids 2007

Analysis Method: SW846-8260B

Prep Method: SW846-5035 | **Instrument:** HP-GC/MS | **Analyst: AC**

Solid Matrix

Compound	Prep Date	Est. Limit (ug/kg)	Spike Conc. (ug/kg)	1	2	3	4	5	6	7	Samples (N)	Mean (ug/kg)	Average %Rec	Deviation (s)	MDL (ug/kg)	Reporting Limit (ug/kg)
				5/2/2007	5/2/2007	5/2/2007	5/2/2007	5/2/2007	5/2/2007	5/2/2007						
Dichlorodifluoromethane**	5/2/2007	5.00	20.00	14.330	13.980	14.530	14.110	14.400	14.290	17.29	7	14.27	71.4%	1.07	3.36	5.00
Chloromethane	5/2/2007	10.00	20.00	14.530	16.610	14.050	14.500	15.250	15.200	20.12	7	15.02	75.1%	1.94	6.10	10.00
Vinyl chloride	5/2/2007	5.00	20.00	15.760	15.730	16.200	15.460	16.380	16.250	14.42	7	15.96	79.8%	0.62	1.95	5.00
Bromomethane	5/2/2007	5.00	20.00	19.910	20.120	20.930	18.500	20.380	19.230	23.55	7	19.85	99.2%	1.49	4.68	5.00
Chloroethane	5/2/2007	5.00	20.00	17.300	16.610	17.150	17.340	17.390	17.260	14.94	7	17.18	85.9%	0.82	2.57	5.00
Trichlorofluoromethane	5/2/2007	5.00	20.00	15.150	14.880	15.600	17.340	14.740	14.850	17.14	7	15.43	77.1%	1.03	3.23	5.00
Acrolein**	5/2/2007	50.00	100.00	149.570	153.310	158.520	140.440	164.570	146.860	124.530	7	148.26	148.3%	12.10	38.03	50.00
1,1-Dichloroethene	5/2/2007	5.00	20.00	16.630	16.930	17.430	16.710	17.950	17.110	19.06	7	17.13	85.6%	0.80	2.50	5.00
Iodomethane	5/2/2007	5.00	20.00	13.980	12.080	14.430	14.120	14.100	14.330	16.65	7	13.84	69.2%	1.23	3.87	5.00
Carbon Disulfide	5/2/2007	5.00	20.00	17.310	17.820	18.500	17.430	18.520	17.980	19.83	7	17.93	89.6%	0.80	2.50	5.00
Methylene Chloride	5/2/2007	5.00	20.00	21.660	21.920	22.740	22.650	24.060	23.280	26.52	7	22.72	113.6%	1.52	4.79	5.00
Acetone	5/2/2007	20.00	40.00	38.370	46.650	43.260	40.080	43.490	49.020	46.28	7	43.48	108.7%	3.49	10.96	20.00
trans-1,2-Dichloroethene	5/2/2007	5.00	20.00	18.380	18.930	19.520	18.620	19.520	18.940	21.04	7	18.99	94.9%	0.82	2.57	5.00
Methyl-tert-Butylether	5/2/2007	5.00	20.00	20.860	21.840	21.890	21.600	22.850	21.690	21.97	7	21.79	108.9%	0.54	1.71	5.00
1,1-Dichloroethane	5/2/2007	5.00	20.00	18.310	18.170	18.370	18.280	18.310	18.250	20.72	7	18.28	91.4%	0.86	2.69	5.00
Acrylonitrile	5/2/2007	50.00	100.00	129.060	146.490	137.950	134.330	144.000	158.990	150.01	7	141.80	141.8%	9.36	29.41	50.00
Vinyl acetate	5/2/2007	20.00	40.00	37.950	39.200	35.220	32.840	33.260	34.700	27.62	7	35.53	88.8%	3.51	11.02	20.00
cis-1,2-Dichloroethene	5/2/2007	5.00	20.00	19.380	19.700	20.000	19.440	20.550	19.770	21.75	7	19.81	99.0%	0.77	2.42	5.00
2,2-dichloropropane	5/2/2007	5.00	20.00	18.540	19.330	20.220	18.700	20.240	19.170	20.51	7	19.37	96.8%	0.73	2.31	5.00
Methyl Ethyl Ketone	5/2/2007	50.00	40.00	38.010	53.310	49.710	49.070	53.550	57.180	53.09	7	50.14	125.3%	5.70	17.90	50.00
Bromochloromethane	5/2/2007	5.00	20.00	18.550	18.950	18.270	17.800	18.910	18.180	19.86	7	18.44	92.2%	0.62	1.96	5.00
Chloroform	5/2/2007	5.00	20.00	18.560	18.900	18.620	18.390	19.320	18.350	21.06	7	18.69	93.5%	0.89	2.78	5.00
1,1,1-Trichloroethane	5/2/2007	5.00	20.00	18.910	19.470	20.640	19.290	20.410	19.540	20.67	7	19.71	98.6%	0.66	2.07	5.00
1,1-Dichloropropene	5/2/2007	5.00	20.00	18.820	19.220	19.990	19.340	20.220	19.230	20.82	7	19.47	97.4%	0.65	2.04	5.00
Carbon Tetrachloride	5/2/2007	5.00	20.00	18.820	19.440	20.290	18.960	20.190	18.740	20.04	7	19.41	97.0%	0.62	1.96	5.00
1,2-Dichloroethane	5/2/2007	5.00	20.00	18.800	19.760	19.380	19.470	20.310	19.750	21.34	7	19.58	97.9%	0.75	2.35	5.00
Benzene	5/2/2007	5.00	20.00	19.800	19.820	20.670	19.740	21.060	20.270	21.64	7	20.23	101.1%	0.67	2.12	5.00
Trichloroethene	5/2/2007	5.00	20.00	19.610	19.300	19.620	18.690	19.180	17.800	20.01	7	19.03	95.2%	0.68	2.13	5.00
1,2-Dichloropropane	5/2/2007	5.00	20.00	20.250	20.530	20.790	20.250	20.810	20.030	21.46	7	20.44	102.2%	0.45	1.40	5.00
Dibromomethane	5/2/2007	10.00	20.00	16.950	22.130	20.870	20.880	18.360	18.350	21.80	7	19.59	98.0%	1.85	5.81	10.00
Bromodichloromethane	5/2/2007	5.00	20.00	19.650	19.990	19.980	19.990	19.510	19.060	20.54	7	19.70	98.5%	0.43	1.36	5.00
cis-1,3-Dichloropropene	5/2/2007	5.00	20.00	19.280	19.850	19.370	19.260	19.150	18.980	19.70	7	19.32	96.6%	0.28	0.89	5.00
MIBK	5/2/2007	5.00	40.00	39.830	43.440	40.750	41.560	42.250	44.680	43.53	7	42.09	105.2%	1.58	4.97	5.00
Toluene	5/2/2007	5.00	20.00	18.010	17.810	17.230	17.230	17.100	16.480	18.88	7	17.31	86.6%	0.72	2.25	5.00
trans-1,3-Dichloropropene	5/2/2007	5.00	20.00	19.620	19.060	18.140	18.730	18.710	18.260	19.96	7	18.75	93.8%	0.62	1.96	5.00
1,1,2-Trichloroethane	5/2/2007	5.00	20.00	19.430	19.420	18.860	18.290	18.580	17.630	19.80	7	18.70	93.5%	0.70	2.20	5.00
Tetrachloroethene	5/2/2007	5.00	20.00	19.720	19.570	18.520	19.460	18.870	17.330	20.71	7	18.91	94.6%	0.99	3.10	5.00
2-Hexanone	5/2/2007	100.00	40.00	38.450	42.460	38.720	91.970	40.240	42.010	41.66	7	48.98	122.4%	18.04	56.69	100.00
Dibromochloromethane	5/2/2007	5.00	20.00	17.780	17.600	17.340	17.550	16.590	16.800	17.78	7	17.28	86.4%	0.44	1.38	5.00
1,2-Dibromoethane	5/2/2007	5.00	20.00	18.090	18.980	17.590	17.960	17.350	17.510	18.65	7	17.91	89.6%	0.56	1.77	5.00
Chlorobenzene	5/2/2007	5.00	20.00	17.160	16.770	16.270	16.060	15.510	15.040	17.45	7	16.14	80.7%	0.81	2.53	5.00
1,1,1,2-Tetrachloroethane	5/2/2007	5.00	20.00	19.250	19.590	19.300	19.070	19.480	18.250	18.85	7	19.16	95.8%	0.42	1.32	5.00
Ethylbenzene	5/2/2007	5.00	20.00	18.770	18.760	19.910	18.500	18.830	18.120	19.45	7	18.82	94.1%	0.55	1.73	5.00
m,p-Xylenes	5/2/2007	5.00	40.00	36.740	36.720	37.350	35.810	36.860	35.220	38.35	7	36.45	91.1%	0.94	2.95	5.00
o-Xylene	5/2/2007	5.00	20.00	18.450	18.330	18.460	17.670	18.180	17.470	18.93	7	18.09	90.5%	0.46	1.45	5.00
Styrene	5/2/2007	5.00	20.00	18.520	18.000	18.120	17.490	17.880	16.920	18.25	7	17.82	89.1%	0.49	1.55	5.00
Bromoform	5/2/2007	5.00	20.00	18.930	19.900	18.560	18.730	18.760	18.370	18.67	7	18.88	94.4%	0.46	1.44	5.00
Isopropyl Benzene	5/2/2007	5.00	20.00	17.990	18.020	18.170	17.320	17.840	16.990	18.37	7	17.72	88.6%	0.45	1.42	5.00
n-Propylbenzene	5/2/2007	5.00	20.00	17.880	17.600	17.970	16.980	17.340	16.690	18.28	7	17.41	87.1%	0.52	1.65	5.00
1,1,2,2-Tetrachloroethane	5/2/2007	5.00	20.00	18.780	18.900	18.300	18.000	18.380	18.570	18.72	7	18.49	92.4%	0.29	0.91	5.00
Bromobenzene	5/2/2007	5.00	20.00	18.110	17.990	17.990	17.530	17.680	17.100	18.50	7	17.73	88.7%	0.42	1.31	5.00
1,3,5-trimethylbenzene	5/2/2007	5.00	20.00	17.720	17.290	17.620	16.860	17.050	16.260	17.91	7	17.13	85.7%	0.53	1.67	5.00
2-Chlorotoluene	5/2/2007	5.00	20.00	17.760	17.740	17.860	17.030	17.390	16.530	18.24	7	17.39	86.9%	0.53	1.67	5.00

VOC MDL Study Solids 2007

Analysis Method: SW846-8260B

Prep Method: SW846-5035 | **Instrument:** HP-GC/MS | **Analyst: AC**

Solid Matrix

Compound	Prep Date	Est. Limit (ug/kg)	Spike Conc. (ug/kg)	1	2	3	4	5	6	7	Samples (N)	Mean (ug/kg)	Average %Rec	Deviation (s)	MDL (ug/kg)	Reporting Limit (ug/kg)
				5/2/2007	5/2/2007	5/2/2007	5/2/2007	5/2/2007	5/2/2007	5/2/2007						
1,2,3-Trichloropropane	5/2/2007	5.00	20.00	19.000	19.990	18.810	16.790	19.270	19.530	19.76	7	18.90	94.5%	0.99	3.10	5.00
4-Chlorotoluene	5/2/2007	5.00	20.00	17.970	17.510	17.860	16.790	17.010	16.200	17.55	7	17.22	86.1%	0.59	1.85	5.00
tert-Butylbenzene	5/2/2007	5.00	20.00	17.000	16.820	17.140	15.930	16.330	15.910	17.05	7	16.52	82.6%	0.49	1.55	5.00
1,2,4-Trimethylbenzene	5/2/2007	5.00	20.00	17.710	17.220	17.260	16.430	16.600	15.920	17.21	7	16.86	84.3%	0.57	1.78	5.00
sec-Butylbenzene	5/2/2007	5.00	20.00	17.340	17.090	17.480	16.440	16.800	15.980	17.75	7	16.86	84.3%	0.57	1.80	5.00
p-Isopropyltoluene	5/2/2007	5.00	20.00	17.260	17.130	17.040	16.120	16.280	15.570	17.04	7	16.57	82.8%	0.60	1.87	5.00
1,3-Dichlorobenzene	5/2/2007	5.00	20.00	17.540	16.940	16.850	16.070	15.860	15.110	16.47	7	16.40	82.0%	0.74	2.33	5.00
1,4-Dichlorobenzene	5/2/2007	5.00	20.00	21.550	21.010	20.540	20.030	20.020	19.230	19.65	7	20.40	102.0%	0.74	2.33	5.00
n-Butylbenzene	5/2/2007	5.00	20.00	20.390	20.110	20.040	19.420	19.230	18.510	19.30	7	19.62	98.1%	0.60	1.89	5.00
1,2-Dichlorobenzene	5/2/2007	5.00	20.00	21.180	20.800	20.490	20.000	19.830	19.100	19.64	7	20.23	101.2%	0.66	2.09	5.00
1,2-Dibromo-3-chloropropane	5/2/2007	5.00	20.00	21.420	24.490	21.930	21.590	22.280	23.170	21.87	7	22.48	112.4%	1.01	3.16	5.00
Hexachlorobutadiene	5/2/2007	5.00	20.00	20.360	20.360	20.250	19.760	19.390	18.100	19.14	7	19.70	98.5%	0.77	2.41	5.00
1,2,4-Trichlorobenzene	5/2/2007	10.00	20.00	20.340	19.630	18.610	17.950	17.290	16.000	15.78	7	18.30	91.5%	1.60	5.03	10.00
Naphthalene	5/2/2007	5.00	20.00	20.840	22.100	20.460	20.180	20.100	19.230	18.83	7	20.49	102.4%	0.99	3.12	5.00
1,2,3-Trichlorobenzene	5/2/2007	5.00	20.00	21.200	19.810	20.130	19.510	18.550	17.470	16.86	7	19.45	97.2%	1.42	4.46	5.00

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/OA/1100

SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 2

SOP Effective Date: 5/31/07

Controlled Copy on Green Paper

Copy No. _____
Approved By_____
Date _____

1.0 Methods

1.1 This SOP meets the requirements of the following methods: EPA 625, SW846 8270C

2.0 Applicable Matrices

2.1 This method applies to a wide variety of environmental matrices, including ground water, aqueous sludges, soils, sediments, acid and caustic liquors, oils, solvents, polymers, and spent carbons and catalysts

3.0 Detection Limits

3.1 Detection limits vary per compound. Detection limit studies are run annually to determine performance. Current detection limit performance and limits can be found with the QA/QC Officer.

4.0 Scope and Application

4.1 This SOP is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. Direct injection of a sample may be used in limited applications. This SOP can determine the compounds found in Table 1. of Method 8270C

4.2 Method 8270C can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivation, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

4.3 The following compounds may require special treatment when being determined by this SOP:

4.3.1 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.

4.3.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

4.3.3 N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.

4.3.4 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

4.3.5 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material

4.3.6 In addition, analytes in Table 1 are flagged when there are limitations caused by sample preparation and/or chromatographic problems

4.4 The estimated quantitation limit (EQL) is the lowest concentration that the laboratory can routinely achieve within precision and accuracy limits. This concentration is selected as the lowest non-zero

standard in the calibration curve and identified as the reporting limit. Table 1 lists the reporting limits for each reportable compound. Reporting limits are proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

5.0 Summary

5.1 This SOP is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. Direct injection of a sample may be used in limited applications

5.2 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation per matrix type (refer to Table 1).

5.3 Sample extract is injected into a gas chromatograph (GC) containing a narrow-bore fused-silica capillary column. The column is temperature-programmed to separate the analytes, and are transported to the mass spectrometer (MS) for detection in a stream of gas

5.4 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or election 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.

6.0 Responsibilities

6.1 Analyst: Personnel experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool. The analyst has demonstrated the ability to generate acceptable results using this SOP or is under the direct supervision of analysts experienced in the use of gas chromatograph/mass spectrometers. Analyst must perform an initial demonstration of capability prior to reporting results.

7.0 Definitions

7.1 IS: Internal standard, a pure analyte(s) added to a solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same solution. The internal standard must be an analyte that is not a sample component.

7.2 Surrogate: A pure analyte, which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction and is measured with the same procedures used to measure other sample components. The purpose of the surrogate is to monitor method performance with each sample.

7.3 LCS: Laboratory control sample, an aliquot of reagent water to which a known amount of method analytes are added. It is analyzed like a sample and used to determine whether the method is in control. LCS samples are produced using the secondary source standard

7.4 SSS: Secondary source standard, solution purchased from a separate supplier than the primary source standards or from the same supplier with a different lot number. Used to validate a new calibration curve.

7.5 CCV: Continuing calibration verification, sample made up at or near the midpoint of the working calibration range. Used to verify whether the calibration curve is still valid. CCV samples are produced using the primary source standard.

7.6 MS/MSD: Matrix spike and matrix spike duplicate, an aliquot of a selected sample is spiked with a known amount of the method analytes. It is analyzed like a sample and used to determine the presence of matrix interferences. These samples are produced using the secondary source standard.

7.7 Blank: An aliquot of reagent water that is treated as a sample and used to determine if method analytes or other interferences are present.

7.8 GC/MS: Gas chromatograph/Mass spectrometer

7.9 SPCC: System performance check compound

7.10 CCC: Calibration compound

7.11 SVOC: Semi-volatile organic compound

7.12 ICV: Initial Calibration Verification

8.0 Interferences

8.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or concentration of the samples and take corrective action to eliminate the problem.

8.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

9.0 Environmental Health and Safety

9.1 This method uses standards and reagents that are extremely hazardous. All sample and standard preparation should be performed in a fume hood. The wearing of lab coat, safety glasses and gloves is highly recommended to avoid exposure to hazardous agents.

9.2 Consult the FIRSTECHNOLOGY Chemical Hygiene Plan and individual MSDS forms to determine the health hazards of specific compounds

10.0 Equipment and Supplies

10.1 Gas chromatograph/mass spectrometer system

10.1.1 Gas Chromatograph: 5890 Series II GC from HP equipped with Electronic Pressure Control (EPC).

10.1.2 Column - DB-5 MS J&W 30M x 0.25 mm x 0.5 µm film thickness fused silica capillary column.

10.1.3 Mass Spectrometer 5972 from HP

10.1.4 GC/MS interface - interface is capillary - direct into the mass spectrometer source.

10.1.5 Data System - Chem Station from HP

10.1.6 Guard Column - 5 meter deactivated guard column from Restek

10.2 Syringe – 5, 10, 25, 50, 100, & 200-µL.

10.3 Volumetric flasks, Class A – Appropriate sizes with ground-glass stoppers.

10.4 Balance – Analytical, capable of weighing 0 001 g.

10.5 Bottles – glass with polytetrafluorethylene (PTFE) – lined screw caps or crimp tops.

11.0 Reagents

11.1 Reagent grade inorganic chemicals shall be used in all tests Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination

11.2 All standards are stored in a freezer at -10°C or less.

11.3 Organic-free reagent water – All references to water in this method refer to organic-free reagent water, defined by ASTM as Type I reagent water

11.4 Acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, and other appropriate solvents – All solvents should be pesticide quality or equivalent

11.5 Stock standard solutions – All stock standard solutions are purchased as certified solutions from various vendors and stored at 4°C as recommended by the manufacturer. Stock standards are replaced after one year. These standards can be purchased from several vendors such as Ultra, Supelco, ECS, and Restek.

11.6 Internal Standard Solutions (ISS) are purchased from ECS or an equivalent supplier at concentration of 4,000 µg/mL.

11.7 Surrogate Standard: Surrogates are purchased from ECS at a concentration of 1,000-2,000 µg/mL.

11.8 Tuning Standard: mix tuning standard is purchased from ECS at a concentration level of 1.00 µg/mL.

11.9 Calibration Standard: 5 to 8 calibration standards are prepared at different concentration levels (see table 11.1 for concentration range). Calibration standards are stored at 4°C and prepared weekly).

11.10 Matrix Spike Standard: Matrix Spike Standards are purchased from ECS at a concentration level of 1 00-2,000 µg/mL.

11.11 Standards and reagents could be purchased from any vendor with the exception of calibration standards and calibration check standards which should be obtained from different sources.

11.12 Daily Tuning Standard: 50 ug/mL

11.13 Daily Calibration Check: 50 ug/mL.

11 14 Internal Standard: each 1 mL sample is spiked with 10 uL of internal standard.

TABLE 11.1: Calibration Standards			
Concentration of Stock Calibration Standard = 200 µg/mL			
Calibration Level	**Concentration**	**Volume of Standard Used (mL)**	**Final Volume using Methylene Chloride ($McCl_2$)**
1	10	25	500
2	20	50	500
3	30	75	500
4	40	100	500
5	50	125	500
6	60	150	500
7	80	200	500
8	100	250	500

Each standard above is spiked with 5 µl of internal standard.

12.0 Sample Collection, Preservation and Handling

12.1 Samples must be collected in pre-cleaned glass containers. Water samples will be collected in 1000 ml amber bottles, and soil in soil jars. Both are kept at 4°C ±2°C.

12.2 Water samples must be extracted within 7 days of sampling, soils within 14 days

12.3 Store sample extracts at -10°C, protected from light, in sealed vials (e.g., screw-cap vials or crimp-capped vials) equipped with un-pierced PTFE-lined septa. Extracts must be analyzed within 40 days

13.0 Quality Control

13.1 A preparation batch of samples is defined as a group of up to twenty field samples of similar matrix type that have been prepared at the same time or time sequence with the same lots of reagents for the same analysis. In addition to the twenty samples, each preparatory batch will contain at a minimum, a method blank, a laboratory control sample, a matrix spike, a matrix spike duplicate and a matrix duplicate. An analytical, or instrumental batch is defined as samples that are analyzed together within the same time period or in continuous sequential time periods.

13.2 The GC/MS system must meet the calibration verification acceptance criteria every 12 hours.

13.2.1 The GC/MS system must be tuned to meet DFTPP criteria listed on Table 14.1. However, other documented tuning criteria may be used provided method performance is not adversely affected (e.g., CLP Method 525, manufacturer's instructions).

13.2.2 The initial calibration of each compound of interest should be verified once every twelve hours prior to sample analysis, using the introduction technique and conditions used for samples. This is done by analyzing a continuing calibration verification sample. This CCV should have a concentration near the midpoint of the calibration range.

13 2 3 A method blank should be analyzed after the calibration standard, or at any other time during the tune shift, to ensure that the system is free of contaminants

13.3 The RRT of the sample component must be within ±0.06 RRT units of the standard component.

13.4 Initial Demonstration of Proficiency – Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix.

13.5 Sample Quality Control for Preparation and Analysis – The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample

13.5.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

13.5 2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate un-spiked sample or on matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an un-spiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

13.5.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight 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

13 5.3.1 The LCS Standard should be a secondary source standard. It should be obtained from a different vendor than the primary source, or from the same vendor provided a different lot number can be documented to be from a different source.

13.5 3.2 The LCS Standard should be prepared as a combined standard including both fractions, even if acid and base/neutral fractions are to be separated for method 625. The LCS should start as if it is an actual sample.

13.6 Surrogate recoveries – The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developing by the laboratory.

13 7 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc

13.8 MDLs are determined in either reagent water or organic-free sand/soil and verified annually. (Project specific requirements may require that the MDL study be performed on the site-specific matrix.)

14.0 Procedure

14.1 Sample Preparation

14.1.1 Samples are normally prepared by one of the following methods prior to GC/MS analysis. Refer to the appropriate SOPs for specific procedures of sample extraction.

Matrix	SOP
Water	SOP/OE/1500
Soil/sediment	SOP/OE/1600
Oil/Solvent (Waste Dilution)	SOP/OE/1700

14.2 Initial calibration to establish the GC/MS operating conditions, using the following recommendations as guidance.

Mass range:	35.0 – 500.0 amu
Scan time:	0.99 scan/sec
Initial temperature:	50°C
Initial time:	1 minute
Rate:	15°/minute
Temperature:	120°C @ 20°C/minute
Temperature:	280°C @ 2.0°C/minute
Final temperature:	300°C
Injector temperature:	250°C
Detector temperature:	290°C
Equilibrium Time:	0.5 minutes
Injection volume:	1µL
Carrier gas:	Helium at approximately 1.22 ml/min

Split injection is allowed if the sensitivity of the mass spectrometer is sufficient.

14.3 1 mL of 50 µg/mL tuning standard is injected and spectra checked for DFTPP mass intensity criteria as per the following table. DDT degradation is inspected and benzidine and pentachlorophenol is checked for tailing.

Table 14.1: DFTPP Time	
MASS	FOR ABUNDANCE CRITERIA
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 70
127	40 – 60% of mass 198
197	< 1% of mass 198
198	Base Peak 100%
199	5 – 9% of 198
275	10 – 30% of mass 198
365	> 1% of mass 198
441	> 40% of mass 198
443	17 – 23% of mass 442

14 4 A set of 5-8 calibration standards at concentrations per Table 11.1 are prepared. Each standard is injected with 10 mL of internal standard. Average RF curve is generated. RSD for all the analytes should be less than 15%. All analytes with an RSD greater than 15% are individually inspected. The inspection includes standard preparation for integration, presence of active sites etc. Certain analytes might need removal of some data points.

14.5 System Performance Check Compounds (SPCCS):

14 5.1 SPCC are a part calibration compound. These compounds listed in the following table should meet the minimum RF criteria of 0.05.

Table 14.2 System Performance Check Compounds (SPCC)
1. N-Nitroso-di-n-propylamine
2. Hexachlorocyclopentadiene
3. 2,4-dinitrophenol
4. 4-nitrophenol

14.6 Calibration Check Compounds (CCC):

14.6.1 The RSD of any CCC are listed in the following table should be less than 30% in the initial calibration curve

Table 14.3 Calibration Check Compound (CCC)	
Base/Neutral Fraction	**Acid Fraction**
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a) pyrene	

14 6 2 The relative retention time of each target analytes in each calibration standard should be within 06 units

14.6 3 GC/MS calibration verification is performed before analysis of samples begins. Verification includes the following steps

14.6.3 1 Fifty (50) ng of tuning standard is injected

14.6.3.2 Fifty (50) ng concentration of calibration check standard is analyzed. If the CCC in this analysis are equal to or less than 20%, then the initial calibration is assumed to be valid. CCC should be checked in 12th analytical shift.

14.7 System Performance Check Compound (SPCC):

14.7 1 SPCC are checked for minimum RF of 0.05 during the 12th analytical shift. If minimum response is met, the system is evaluated and corrective action, including column cut, injection line & deactivation is taken

14.7.1.1 Method blank or solvent blank is analyzed after the CCC run to limit the carryover of the contamination to the sample.

14.8 Calibration Check Compound (CCC):

14.8.1 If the percent difference for each CCC is less than or equal to 20% then initial calibration is valid and sample analysis can proceed. If the CCC fails the criteria, then CCC is evaluated via manual quantitation and standard is reanalyzed if needed.

14.9 Internal Standard Retention Time:

14.9.1 The RT of internal standard ~ CCC are evaluated and any change more than 30 seconds from the mid level standard of initial calibration is evaluated. Column cut and change of guard column will result in change of RT. In that case compensation is made and method is updated with new RT.

14.10 Internal Standard Response:

14.10.1 Area of internal standards ~ CCC should be -50% to +100% of that mid level standard from the initial calibration curve. If this criteria is not met then system s inspected and corrections made as needed

14.11 GC/MS Analysis of Sample

14.11.1 Each mL. extract of sample is spiked with 10 uL of internal standard before analysis. Any analyte outside the calibration range results in reanalysis after dilution of the sample. All conditions used in initial calibration are used for sample analysis

14.12 Qualitative Identification

The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. All hits should be visually compared to the reference spectrum for confirmation. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met

14.12.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion

14.12.2 The RRT of the sample component is within ±0.06 RRT units of the RRT of the standard component.

14.12.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

14.12.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights Otherwise, structural isomers are identified as isomeric pairs

14.12.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds When analytes co-elute (i e , only one chromatographic peak is apparent), the identification criterion can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound

14.12.6 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. When serving the role of QA (or referee) laboratory, tentatively identified compounds (TICs) shall always be reported Guidelines for making tentative identification are:

14.12.6.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

14.12.6.2 The relative intensities of the major ions should agree within ± 30% to be consistent with target compound list identification (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 20 and 80%).

14.12.6.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

14.12.6.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds

14.12.6.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

14.12.6.6 Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification

14.13 Quantitative analysis

14.13.1 When a compound has been identified, the quantitation of that compound is based on the integrated abundance from the EICP of the primary characteristic ion Quantitation is accomplished using the internal standard technique, as described in sections 15.5 and 15.6. The internal standard used must be the one nearest the retention time of that of a given analyte

14.13.2 Sediment/soil samples are reported on a dry weight basis, while sludges and wastes are reported on a wet weight basis. The percent dry weight of the sample (see section 15.7) must be reported along with the data in either instance.

14.14

15.0 Calculations

15.1 Calculation of response factors:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate
A_{is} = Peak area (or height) of the internal standard
C_s = Concentration of the analyte or surrogate, in ug/L
C_{is} = Concentration of the analyte or internal standard, in ug/L.

15.2 Average value

$$\overline{X} = \frac{1}{n}\sum X$$

where:

X = value to be averaged
n = the number of values
$\overline{X}$ = the average

15.3 Calculation of % Difference

$$\%\,\text{Difference} = \frac{RF_v - \overline{RF}}{\overline{RF}} \times 100$$

where:

RF_v = Response factor from verified standard
$\overline{RF}$ = Mean response factor from the initial calibration.

15.4 Sample concentration for liquid (ug/L) and soil samples (ug/Kg)

$$\text{Concentration} = \frac{\left[\dfrac{A_x \times C_{is}}{\overline{CF} \times A_{is}}\right]}{S} \times D$$

where:

A_x = Response for the analyte in the sample, in area counts
D = Dilution factor
$\overline{CF}$ = Mean CF value from the initial calibration, in counts per ng

C_{is} = Concentration of internal standard in ug/mL
A_{is} = Area count of associated internal standard
S = Weight or volume of sample

15.5 Calculation of Recovery

Surrogates:

$$\%\text{Recovery} = \frac{C_m}{C_s} \times 100$$

where:

C_m = Measured concentration of the surrogate
C_s = Nominal (or theoretical) Concentration of the surrogate

MS/MSD and LCS analyses:

$$\%\text{Recovery} = \frac{C_s - C_u}{C_n} \times 100$$

where:

C_s = Measured concentration of the spiked sample aliquot
C_u = Measured concentration of the un-spiked sample aliquot (use 0 for LCS analyses)
C_n = Nominal (or theoretical) concentration of the spiked sample aliquot

15.6 Calculation of Precision

$$\text{RPD} = \frac{(C_1 - C_2)}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where:

C_1 = Measured concentration of the first sample
C_2 = Measured concentration of the second sample

15.7 Best-fit line (linear regression)

15.7.1 Step 1; minimize the sum of the squares

$$S_{xx} = \sum \left(x_i - \bar{x}\right)^2$$

where:

S_{xx} = sum of squares for concentration of standard solution
X_i = individual data points for concentration
$\overline{X}$ = average value for concentrations, see 10.2

$$S_{yy} = \sum \left(y_i - \bar{y}\right)^2$$

where:

S_{yy} = sum of squares for area of a peak
Y_i = individual data points for area of peak

$\overline{Y}$ = average value for area of peaks, see 10.2

$$S_{xy} = \sum (X_i - \overline{X})(Y_i - \overline{Y})$$

where:

S_{xy} = sum of squares for concentration of standard solution
X_i = individual data points for concentration
$\overline{X}$ = average value for concentrations, see 10.2
Y_i = individual data points for area of peak
$\overline{Y}$ = average value for area of peak, see 10.2

15.7.2 Step 2; calculate the slope and intercept

$$m_{LS} = \frac{S_{xx}}{S_{yy}}$$

where:

S_{xx} = sum of squares for concentration of standard solution
S_{yy} = sum of squares for area of a peak
m_{LS} = least-squares slope

$$b_{LS} = \overline{Y} - m_{LS}\overline{X}$$

where:

$\overline{Y}$ = average value for area of peak
$\overline{X}$ = average value for concentration
m_{LS} = least-squares slope
b_{LS} = least-squares intercept

15.7.3 Step 3; calculate the best-fit line

$$Y = m_{LS}X + b_{LS}$$

where:

Y = calculated peak areas
m_{LS} = least-squares slope
b_{LS} = least-squares intercept
X = concentrations

16.0 Data Validation

16.1 All data must have been acquired during an accepted 12-hour tune shift. If the sample was not injected during the twelve-hour tune shift then it must be reanalyzed within an acceptable tune shift

16.2 The initial calibration must consist of at least five points, with the low standard being near but above the MDL.

16.2.1 The average percentage RSD for the compound RRF's must be <15%. If the RSD for the response factor is not less than 15% then linear regression may be used for that analyte. The correlation coefficient must be at least 0.990 or higher to use linear regression. If these requirements cannot be met then that particular analyte is not reported or the entire calibration must be repeated.

16.2.2 The ICV must be within 15% of actual concentration. If the ICV is not within 15% then the calibration must be repeated. It is recommended to clean the injection port and remove 6-12 inches of the column. Prepare and analyze fresh standards.

16.3 The minimum acceptable RRF for the SPCC's is 0.050. If this criterion is not met, the system must be evaluated and corrective action taken before sample analysis begins. Corrective action would include cleaning the injection port, breaking off 6-12 inches from the front of the column and recalibrating.

16.4 The percent relative standard deviation (%RSD), of the CCC's must be <30% If not corrective action must be taken before sample analysis begins. Corrective action includes cleaning the injection port, breaking off 6-12 inches from the front of the column, preparing fresh standards, and recalibrating.

16.5 The relative retention times (RRT) of each target analyte in each calibration standard must be within 0.06 RRT units. If not check for loss of helium pressure, leaks, proper split flow, total flow, and recalibrate.

16.6 Surrogate recoveries must be within established laboratory control limits. Refer to Table A1 below for estimated upper and lower control points for surrogate sample results. Check the surrogate calculations for correctness for all samples, blanks, LCS, MS, and their duplicates. Check that the surrogate recoveries are properly control-charted for the LCS and method blank(s) The following acceptance criteria apply to surrogate recoveries.

- The surrogate recoveries for all field and QC samples must be within control limits on control charts. If the LCS and/or method blank recoveries are outside the limits, reanalysis must be performed for verification If still outside QC control limits, all associated QC and field samples must be re-extracted.

- Sample surrogate recoveries are compared to the LCS surrogate control limits. If sample surrogate recoveries exceed these limits, all associated QC and field samples must be re-extracted. If the results are still outside the limits, then the project manager is to be contacted to discuss further alternatives and the impact on the project

- Even though control charts must be maintained for the LCS, the acceptance criteria derived from these charts may not be appropriate if the charted ranges are too wide. A maximum default range should be used This range should also be compared to the guidance limits as stated in the method.

- RPD of LCS duplicates should be ≤20%.

16.7 Laboratory control samples/matrix spike samples must have recoveries within established laboratory guidelines Refer to Table A1 below for estimated upper and lower control points for LCS and MS sample results

- Assess that LCSs were prepared at the required frequency. Evaluate the target compounds using control charts, where applicable. If all target compounds are not within control limits, the LCS is to be reanalyzed for verification. If it is still out of control limits, then all field and QC samples in the batch must be re-extracted and analyzed.

- The RPD of inter-batch LCSs should fall within the control limits determined from the precision control charts. However, if the RPD is outside these control limits, the batch will not be rejected, as long as the LCS recovery is acceptable. This precision information should be evaluated to see if systematic problems could be identified. If problems are suspected, the method should be fully evaluated.

16.8 Assess that matrix spike/matrix spike duplicates were analyzed at required frequency. The analyst shall also verify that the samples were spiked at the appropriate level.

16.8.1 If the target analyte concentrations are known, spike to increase the background concentration by a factor of approximately two.

16.8.2 If an action level exists, spike at this level.

16.8.3 If neither of the first two conditions applies, spike at a level between the low and mid-level calibration standards.

16.9 Acceptance criteria are that all percentage Recovery and/or RPD results meet project-established goals. If no project goals are specified, then results must be within the indicated control limits on the appropriate LCS control charts. Percent recoveries for LCS are provided on Table A1. If these conditions are not met, perform the following corrective actions as appropriate.

16.9.1 If both LCS and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be re-extracted and analyzed.

16.9.2 If the MS/MSD is unacceptable, but the LCS is acceptable, then a potential matrix effect has been identified. Reanalyze to verify matrix effect. If a matrix effect is still suspected, then the project manager must be contacted to discuss further alternatives and the potential impact on the project. Reported data should be flagged. Reasonable attempts must be made to address matrix interference.

16.10 Assess that matrix duplicates were analyzed at required frequency. Acceptance criteria are that all RPD results meet project-established goals. Percent recoveries for MS are provided on Table A1. If these goals are not met, perform the following corrective actions, as appropriate.

16.10.1 Reanalyze the matrix duplicates to verify a matrix effect.

16.10.2 If the duplicate precision is still unacceptable, then a potential matrix effect has been identified. The project manager must be contacted to discuss further alternatives and the potential impact on the project.

16.11 Internal standard areas must meet acceptance criteria of 50% - 200% of their theoretical value to be accepted. If IS values fall outside this range then the sample must be rerun to confirm the failure. Check for air leaks as they cause a loss in sensitivity. If a leak is found recalibrate and rerun the sample. If no leak is present, corrective action can also include diluting the sample to reduce the concentration of interfering compounds causing low/high recovery. If this does not bring IS values within range then the sample must be re-extracted.

16.12 Assess the method blanks. The analyst shall confirm that this blank was analyzed at the required frequency. The method blank should not exhibit any contamination of any analyte above the MDL for any of the method target analytes.

- Corrective action should be performed any time method target analytes are detected above the MDL to reduce and control contamination

- Corrective action will be required if site-specific target analytes are detected at greater than 5% of the regulatory limit for that analyte or if the concentration in the blank is greater than 5% of that sample, or if any method target analyte is detected above the low standard used during initial calibration

16.12.1 First, assess the effect of the contaminants on the samples

16.12.2 Corrective action would include re-extraction and analysis of all field and QC samples in the batch if some or all of the samples also contained levels of target analytes that exceeded the above criteria

16.12.3 If none of the field samples had values above the stated criteria, then reanalysis may not be necessary

16.12.4 The source of contamination should still be investigated, and when possible reduced or eliminated. Any time contamination is noted in the method blank, the situation and impact on the data should be discussed in the case narrative.

16.13 The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot

17.0 Waste Disposal and Pollution Prevention

17.11 Refer to the laboratory waste disposal SOP A-2050 for specific guidelines for disposing of waste.

17.12 Pollution prevention is any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. This procedure generates solvent waste and uses solvent based standards. The laboratory should minimize the amount of standards and solvents used whenever possible. Small volumes of standards are used to create calibration and QC standards. Concentrated standards should be ordered in quantities sufficient to be fully utilized by their expiration date, to eliminate unnecessary disposal of concentrated metals standards

18.0 References

18.1 Test Methods For Evaluating Solid Wastes, SW-846, Third Edition, Revision 3, December 1996, Method 8270.

18.2 EPA Method 625: 40 CFR Ch1 Pt 136 App. A

APPENDIX

TABLE A1
CONTROL POINTS FOR LCS/MS AND SURROGATE PERCENT RECOVERIES

Percent Recovery	Water		Soil	
LCS/MS	Lower	Upper	Lower	Upper
N-Nitrosodimethylamine	35	61	57	102
Pyridine	0	48	7	51
Phenol	22	40	64	108
bis (2-chloroethyl) ether	33	110	62	95
2-Chlorophneol	64	96	74	95
1,3-Dichlorobenzene	39	86	66	92
1,4-Dichlorobenzene	41	85	64	97
2-Methylphenol	59	88	62	110
1,2-Dichlorobenzene	41	91	64	100
bis (2-chloroisopropyl) ether	55	109	47	110
3,4-Methylphneol	51	85	72	97
N-Nitroso-di-n-propylamine	55	119	59	108
Nitrobenzene	59	117	73	107
Hexachloroethane	35	87	66	95
Isophorone	69	118	75	110
2,4-Dimethylphenol	49	113	67	105
2-Nitrophenol	70	113	84	109
bis (2-chloroethoxy) methane	69	116	75	105
2,4-Dichlorophenol	73	108	71	115
1,2,4-Trichlorobenzene	54	100	62	107
Naphthalene	58	100	64	101
4-Chloroaniline	64	114	61	120
Hexachlorobutadiene	48	103	62	114
4-chloro-3-methyl phenol	77	109	86	108
2-Methylnaphthalene	59	106	63	109
Hexachlorocyclopentadiene	1	145	55	115
2,4,6-Trichlorophneol	69	125	66	128
2,4,5-Trichlorophenol	68	130	65	132
2-Chloronaphthalene	58	120	62	114
2-Nitroaniline	75	131	79	128
Dimethyl phthalate	67	121	62	121
2,6-Dinitrotoluene	81	115	75	128
Acenapthylene	60	114	57	117
3-Nitroaniline	76	122	80	122
2,4-Dinitrophenol	0	176	37	144
4-Nitrophenol	0	144	64	132
Acenaphthene	58	117	51	113
2,4-Dinitrotoluene	76	126	75	127
Dibenzofuran	51	131	59	110
Diethyl phthalate	66	121	65	118
4-Chlorophenyl phenyl ether	55	132	62	119
4-Nitroaniline	82	117	79	116

TABLE A1 (cont.)
CONTROL POINTS FOR LCS/MS AND SURROGATE PERCENT RECOVERIES

Percent Recovery	Water		Soil	
LCS/MS (CONTINUED)	Lower	Upper	Lower	Upper
Fluorene	36	143	47	117
4,6-Dinitro-2-methyl phenol	0	194	73	135
Diphenylamine	67	119	62	119
1,2-Diphenylhydrazine	66	131	74	104
4-Bromophenyl phenyl ether	57	143	61	128
Hexachlorobenzene	70	135	63	129
Pentachlorophenol	13	147	55	125
Phenanthrene	64	128	62	116
Anthracene	64	128	61	115
Carbazole	60	127	62	120
Di-n-butyl phthalate	55	137	64	120
Fluoranthene	60	140	60	115
Brenzidine	0	74	0	81
Pyrene	69	121	55	131
Butyl benzyl phthalate	60	121	68	118
bis (2-ethylhexyl) phthalate	58	123	64	122
3,3'-Dichlorobenzidine	59	127	64	115
Benzo [a] anthracene	65	130	62	119
Chrysene	66	133	63	119
Di-n-octyl phthalate	60	126	71	124
Benzo [b] fluoranthene	66	126	71	107
Benzo [k] fluoranthene	64	138	71	123
Benzo [a] pyrene	77	125	81	112
Indeno [1,2,3-cd] pyrene	73	122	78	118
Dibenzo [a,h] anthracene	74	120	81	116
Benzo [g,h,i] perylene	73	119	85	113
Surrogate				
2-Fluorophenol	21	100	25	121
Phenol-d6	10	94	24	113
nitrobenzene-d5	35	114	23	120
2-Fluorobiphenyl	43	116	30	115
2,4,6-Tribromophenol	10	123	19	122
Terphenyl-d14	33	141	18	137

Table A2

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES

ASSIGNED FOR QUANTITATION

1,4-Dischlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis (2-chloroethyl) ether	Bis (2-chloroethoxy) methane	1-Chloronaphthalene
Bis (2-chloroisopropyl)	4-Chloroanline	2-Chloronaphthalene
Ether	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
2-Chlorophenol	2,4-Dichlorophenol	Dibenzofuran
1,3-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,4-Dichlorobenzene	cx, cx-Dimethyl-phenethylamine	Dimethyl phthalate
1,2-Dichlorobenzene	2,4-Dimethyphenol	2,4-Dinitrophenol
Ethy methanesulfonate	Hexachlorobutadiene	2,4-Dinitrotoluene
2-Fluorophenol (surr)	Isophorone	2,6-Dinitrotoluene
Hexachloroethane	1-Methylnapthalene	Fluorene
Methyl methanesulfonate	2-Methylnaphthalene	2-Fluorobiphenyl (surr)
2-Methylphenol	Naphthalene	Hexachlorocyclopentadiene
4-Methylphenol	Nitrobenzene	1-Naphthylamine
N-Nirosodimethylamine	Nitrobenzene-d8 (surr)	2-Naphthylamine
N-Nitrosodi-n-propylamine	2-Nitrophenol	2-Nitroaniline
Phenol	N-Nitrosodi-n-butylamine	3-Nitroaniline
Phenol-d6 (surr)	N-Nitrosopiperidine	4-Niroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitrophenol
Pyridine		Pentachlorobenzene
		1,2,4,5-Tetra-chlorobenzene
		2,3,4,6-Tera-chlorophenol
		2,4,6-Tribromo-phenol (surr)
		2,4,5-Trichloro-phenol
Phenanthrene-d10	**Chrysene-d12**	**Perylene-d12**
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl)phthalate	Benzo(g,h,i)-perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
Carbazole	Chrysene	Dibenz(a,j)acridine
4,6-Dinitro-2-methyla-phenol	3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene
Diphenylamine	p-Dimethylaminoazobenzene	
Fluoranthene	Pyrene	
Hexachlorobenzene	Terphenyl-d14 (surr)	
N-Nitrosodipheylamine	7,12-Dimethylbenz-(a)anthracene	
Pentachlorophenol	Di-n-octyl phthalate	
Pentachloronitrobenzene	Indeno)1,2,3-cd) pyrene	
Phenacetin	3-Methylcholanthrene	
Phenanthrene		
Pronamide		

(surr) = surrogate

MDL 2007 SVOC Liquids

Analytical Method: SW846 8270C

Extraction Method: SW846 3510C

			Replicate Number												
			1	2	3	4	5	6	7						
		Extract Date	3/19/2007	3/19/2007	3/19/2007	3/21/2007	3/26/2007	3/26/2007	4/4/2007						
		Analysis Date	4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/12/2007						Reporting
	Spike Conc	Est. Limit								Samples	Mean	Average	Deviation	MDL	Limit
Compound	(ug/L)	(ug/L)								(N)	(ug/L)	%Rec.	(s)	(ug/L)	(ug/L)
2-Ethoxyethanol	10.00	10	1.07	1.05	0.70	0.57	0.51	0.75	0.95	7	0.80	8.00%	0.23	0.71	10
Pyridine**	10.00	10	7.87	7.74	5.64	6.16	5.34	6.67	8.84	7	6.89	68.9%	1.29	4.06	10
N-Nitrosodimethylamine	10.00	10	4.76	4.93	4.65	4.57	4.52	4.17	5.49	7	4.73	47.3%	0.41	1.29	10
Aniline	10.00	10	4.80	6.48	3.86	1.53	5.21	4.46	5.97	7	4.62	46.2%	1.62	5.10	10
Phenol	10.00	10	8.98	9.49	6.77	3.58	4.10	3.29	4.24	7	5.78	57.8%	2.62	8.24	10
Cyclohexanone	10.00	10	5.41	6.89	6.22	4.39	4.57	4.45	5.54	7	5.35	53.5%	0.96	3.01	10
bis(2-Chloroethyl)ether	10.00	10	6.34	7.70	7.05	5.80	7.22	5.32	7.12	7	6.65	66.5%	0.86	2.69	10
2-Chlorophenol	10.00	10	5.71	6.62	6.10	6.34	7.29	5.50	7.38	7	6.42	64.2%	0.73	2.29	10
1,3-Dichlorobenzene	10.00	10	1.44	2.16	1.40	2.59	3.36	2.83	4.29	7	2.58	25.8%	1.04	3.26	10
1,4-Dichlorobenzene	10.00	10	1.37	2.51	1.33	2.88	3.54	2.68	4.58	7	2.70	27.0%	1.15	3.62	10
Benzyl alcohol	10.00	10	6.24	7.59	7.02	6.58	7.50	5.97	8.11	7	7.00	70.0%	0.78	2.45	10
1,2-Dichlorobenzene	10.00	10	2.03	2.89	1.97	3.07	3.78	3.31	4.98	7	3.15	31.5%	1.04	3.27	10
2-Methylphenol	10.00	20	11.59	12.93	6.74	5.66	6.66	4.36	6.48	7	7.77	77.7%	3.20	10.04	20
bis(2-Chloroisopropyl)ether	10.00	10	5.93	7.45	6.21	5.61	6.83	5.46	6.85	7	6.33	63.3%	0.73	2.30	10
3&4-Methylphenol	10.00	20	11.42	12.77	7.56	4.85	5.77	4.07	5.77	7	7.46	74.6%	3.36	10.57	20
N-Nitroso-di-n-propylamine	10.00	10	7.39	8.72	7.65	7.33	8.33	6.59	8.96	7	7.85	78.5%	0.85	2.67	10
Hexachloroethane	10.00	10	1.07	1.38	0.88	2.15	3.11	2.77	4.11	7	2.21	22.1%	1.19	3.74	10
Nitrobenzene	10.00	10	6.44	7.96	7.01	6.10	7.50	5.73	8.05	7	6.97	69.7%	0.91	2.87	10
Isophorone	10.00	10	6.41	7.68	6.82	6.08	6.95	5.73	7.20	7	6.70	67.0%	0.67	2.11	10
2-Nitrophenol	10.00	10	5.38	6.59	5.94	5.46	7.21	5.43	6.97	7	6.14	61.4%	0.78	2.44	10
2,4-Dimethylphenol	10.00	10	7.25	8.09	1.39	5.15	5.06	0.73	2.36	7	4.29	42.9%	2.87	9.01	10
Benzoic acid	10.00	10	5.55	6.29	4.02	0.84	0.31	1.39	2.28	7	2.95	29.5%	2.36	7.41	10
bis(2-Choloroethoxy)methane	10.00	10	6.61	8.21	7.14	6.38	7.46	5.67	7.84	7	7.04	70.4%	0.88	2.78	10
2,4-Dichlorophenol	10.00	10	5.64	6.78	5.99	5.65	6.75	5.58	6.82	7	6.17	61.7%	0.59	1.84	10
1,2,4-Trichlorobenzene	10.00	10	13.03	16.40	12.61	11.38	13.67	15.12	18.94	7	14.45	144.5%	2.58	8.10	10
Naphthalene	10.00	10	5.33	7.11	5.52	4.54	5.70	5.02	6.85	7	5.72	57.2%	0.94	2.95	10
4-Chloroaniline	10.00	10	4.53	5.88	3.58	4.05	5.81	0.99	1.32	7	3.74	37.4%	1.96	6.16	10
Hexachlorobutadiene	10.00	10	0.80	0.89	0.55	2.20	2.76	1.98	2.91	7	1.73	17.3%	0.97	3.06	10
4-Chloro-3-methylphenol	10.00	10	6.33	7.40	6.05	6.65	7.37	6.15	7.96	7	6.84	68.4%	0.74	2.31	10
2-Methylnaphthalene	10.00	10	3.52	4.84	3.66	4.34	5.13	4.45	5.86	7	4.54	45.4%	0.82	2.58	10
Hexachlorocyclopentadiene	10.00	10	9.00	9.14	9.16	9.86	10.24	10.23	11.08	7	9.82	98.2%	0.76	2.40	10
2,4,6-Trichlorophenol	10.00	10	5.66	7.02	6.16	6.07	6.79	5.72	7.36	7	6.40	64.0%	0.66	2.08	10
2,4,5-Trichlorophenol	10.00	10	6.07	7.13	6.07	5.92	7.06	5.71	7.44	7	6.49	64.9%	0.70	2.19	10
2-Chloronaphthalene	10.00	10	3.57	4.76	3.74	4.79	5.54	4.30	6.03	7	4.68	46.8%	0.90	2.82	10
2-Nitroaniline	10.00	10	6.60	8.08	7.45	6.42	7.68	5.67	7.74	7	7.09	70.9%	0.87	2.75	10
Dimethylphthalate	10.00	10	3.86	5.17	3.07	0.29	0.35	0.46	0.92	7	2.02	20.2%	1.99	6.26	10
Acenaphthylene	10.00	10	5.19	6.50	5.74	5.37	6.10	5.07	6.68	7	5.81	58.1%	0.64	2.01	10
2,6-Dinitrotoluene	10.00	10	6.58	7.85	7.16	6.35	6.94	5.78	6.99	7	6.81	68.1%	0.66	2.06	10
3-Nitoraniline	10.00	10	8.11	9.70	7.75	6.94	8.77	4.65	5.70	7	7.37	73.7%	1.75	5.51	10
Acenaphthene	10.00	10	4.76	6.21	5.45	5.35	6.05	5.05	6.49	7	5.62	56.2%	0.64	2.01	10
2,4-Dinitrophenol	10.00	10	0.00	0.00	2.35	1.67	1.40	1.87	2.93	7	1.46	14.6%	1.11	3.50	10
4-Nitrophenol	10.00	10	1.63	1.89	1.99	2.06	2.22	2.60	3.49	7	2.27	22.7%	0.62	1.94	10
Dibenzofuran	10.00	10	4.70	5.81	4.83	5.58	6.15	4.88	6.37	7	5.47	54.7%	0.68	2.13	10
2,4-Dinitrotoluene	10.00	10	6.14	7.47	7.13	6.23	6.62	5.54	7.04	7	6.60	66.0%	0.67	2.11	10
Diethylphthalate	10.00	10	5.82	7.22	5.81	0.58	0.69	0.77	1.11	6	3.14	31.4%	2.98	9.36	10
4-Chlorophenyl phenylether	10.00	10	4.18	5.04	4.16	5.27	5.90	4.50	6.01	7	5.01	50.1%	0.77	2.41	10
Fluorene	10.00	10	5.49	6.70	5.74	6.14	6.79	5.44	6.88	7	6.17	61.7%	0.63	1.97	10
4-Nitroaniline	10.00	10	5.10	6.16	4.70	4.51	5.45	3.31	4.56	7	4.83	48.3%	0.89	2.79	10
1,2-Diphenylhydrazine	10.00	10	6.94	8.06	6.85	6.88	7.89	6.55	7.93	7	7.30	73.0%	0.63	1.99	10
Benzidine**	50.00	50	59.41	68.86	58.49	64.05	61.62	65.74	59.01	7	62.45	124.9%	3.91	12.30	50
4,6-Dinitro-2-methylphenol	10.00	10	2.65	3.18	3.77	3.28	3.60	3.21	3.99	7	3.38	33.8%	0.44	1.40	10
N-Nitrosodiphenylamine	10.00	10	9.82	11.97	9.86	9.67	10.67	7.75	10.06	7	9.97	99.7%	1.26	3.97	10
4-Bromophenyl phenylether	10.00	10	4.32	5.05	4.25	5.15	5.93	4.46	5.85	7	5.00	50.0%	0.70	2.20	10
Hexachlorobenzene	10.00	10	3.52	4.28	3.45	5.53	5.73	4.07	5.20	7	4.54	45.4%	0.94	2.97	10

MDL 2007 SVOC Liquids

Analytical Method: SW846 8270C

Extraction Method: SW846 3510C

			Replicate Number												
			1	2	3	4	5	6	7						
		Extract Date	3/19/2007	3/19/2007	3/19/2007	3/21/2007	3/26/2007	3/26/2007	4/4/2007						
		Analysis Date	4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/12/2007						Reporting
	Spike Conc	Est. Limit								Samples	Mean	Average	Deviation	MDL	Limit
Compound	(ug/L)	(ug/L)								(N)	(ug/L)	%Rec.	(s)	(ug/L)	(ug/L)
Pentachlorophenol	10.00	10	3.15	3.63	3.68	3.81	3.90	4.08	5.24	7	3.93	39.3%	0.65	2.03	10
Phenanthrene	10.00	10	5.70	6.96	5.80	6.17	6.59	5.54	6.80	7	6.22	62.2%	0.57	1.78	10
Anthracene	10.00	10	4.90	5.97	4.89	5.62	6.30	4.68	5.90	7	5.47	54.7%	0.64	2.00	10
Di-n-butylphthalate	10.00	10	6.18	7.55	6.08	2.77	2.95	2.60	3.29	7	4.49	44.9%	2.04	6.43	10
Fluoranthene	10.00	10	5.60	6.73	5.79	6.03	6.43	5.00	6.27	7	5.98	59.8%	0.58	1.81	10
Pyrene	10.00	10	6.33	7.56	6.83	6.39	7.11	5.42	6.89	7	6.65	66.5%	0.68	2.15	10
Butylbenzylphthalate	10.00	10	5.17	6.38	4.58	3.36	3.34	3.25	4.13	7	4.32	43.2%	1.16	3.65	10
3,3'-Dichlorobenzidine**	50.00	10	21.07	23.59	21.26	20.40	18.96	24.15	21.46	7	21.56	43.1%	1.79	5.63	10
Benzo(a)anthracene	10.00	10	6.25	7.41	6.79	6.21	6.53	5.47	6.68	7	6.48	64.8%	0.60	1.88	10
Chrysene	10.00	10	6.66	7.98	7.14	6.50	7.01	5.80	6.97	7	6.87	68.7%	0.67	2.09	10
bis(2-Ethylhexyl)phthalate	10.00	10	7.84	9.26	8.12	6.78	7.40	7.92	8.20	7	7.93	79.3%	0.76	2.40	10
Di-n-octylphthalate	10.00	10	7.02	8.57	7.58	6.39	7.07	5.83	7.42	7	7.13	71.3%	0.88	2.76	10
Benzo(b)fluoranthene	10.00	10	6.91	8.72	7.55	6.61	7.42	6.20	7.82	7	7.32	73.2%	0.84	2.63	10
Benzo(k)fluoranthene	10.00	10	7.92	9.36	8.71	7.42	7.90	7.18	8.51	7	8.14	81.4%	0.76	2.40	10
Benzo(a)pyrene	10.00	10	6.08	7.26	6.17	6.12	6.50	5.07	6.51	7	6.24	62.4%	0.66	2.07	10
Indeno(1,2,3-cd)pyrene	10.00	10	6.46	7.83	7.10	6.15	6.76	5.92	7.30	7	6.79	67.9%	0.67	2.11	10
Dibenz(a,h)anthracene	10.00	10	6.28	7.83	7.03	5.88	6.38	5.49	6.83	7	6.53	65.3%	0.78	2.44	10
Benzo(g,h,i)perylene	10.00	10	7.45	9.11	8.04	6.97	7.66	6.53	8.16	7	7.70	77.0%	0.84	2.65	10

**Analytes extracted on 4/26/07; analyzed MDL 1,2,3 on 4/27/07, analyzed MDL 4,5,6,7 on 4/28/07

MDL 2007 SVOC Solids

Analytical Method: SW846 8270C															
Extraction Method: SW846 3550B															
			Replicate Number												
			1	2	3	4	5	6	7						
	Extract Date		3/19/2007	3/19/2007	3/19/2007	3/21/2007	3/26/2007	3/26/2007	4/4/2007						
	Analysis Date		4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/12/2007						Reporting
	Spike Conc	Est. Limit								Samples	Mean	Average	Deviation	MDL	Limit
Compound	(ug/kg)	(ug/kg)								(N)	(ug/kg)	%Rec.	(s)	(ug/kg)	(ug/kg)
2-Ethoxyethanol	330.00	1650	115.67	123.67	17.33	866.33	55.33	45.00	17.67	7	177.29	53.72%	306.84	964.39	1650
Pyridine**	330.00	330	87.00	23.67	119.67	24.67	97.67	0.00	21.67	7	53.48	16.2%	46.63	146.57	330
N-Nitrosodimethylamine	330.00	330	170.33	226.67	15.00	27.33	57.00	16.67	16.33	7	75.62	22.9%	86.73	272.58	330
Aniline	330.00	330	175.33	25.67	12.67	7.67	70.67	12.33	10.00	7	44.90	13.6%	61.58	193.54	330
Phenol	330.00	330	254.67	255.67	155.67	163.67	235.67	116.33	128.00	7	187.10	56.7%	60.09	188.86	330
Cyclohexanone	330.00	330	153.00	199.67	27.67	44.67	68.33	30.33	108.33	7	90.29	27.4%	66.21	208.09	330
bis(2-Chloroethyl)ether	330.00	330	209.00	244.33	57.33	86.33	119.33	56.33	45.33	7	116.86	35.4%	79.58	250.13	330
2-Chlorophenol	330.00	330	205.33	229.00	86.00	113.33	155.00	73.33	66.00	7	132.57	40.2%	65.29	205.20	330
1,3-Dichlorobenzene	330.00	330	155.00	205.00	14.67	33.00	44.33	24.67	19.33	7	70.86	21.5%	76.55	240.59	330
1,4-Dichlorobenzene	330.00	330	157.00	213.00	19.00	37.33	49.67	29.33	23.33	7	75.52	22.9%	77.16	242.50	330
Benzyl alcohol	330.00	330	256.33	244.33	169.00	178.00	248.67	121.33	158.33	7	196.57	59.6%	52.91	166.29	330
1,2-Dichlorobenzene	330.00	330	171.33	214.00	26.33	48.33	65.00	36.00	26.67	7	83.95	25.4%	76.46	240.31	330
2-Methylphenol	330.00	330	242.00	251.00	149.33	166.33	237.00	108.33	131.67	7	183.67	55.7%	58.65	184.34	330
bis(2-Chloroisopropyl)ether	330.00	330	221.33	251.00	65.67	90.00	143.67	66.00	55.33	7	127.57	38.7%	80.14	251.89	330
3&4-Methylphenol	330.00	330	235.00	232.67	159.00	167.67	232.33	115.33	153.67	7	185.10	56.1%	48.00	150.86	330
N-Nitroso-di-n-propylamine	330.00	330	293.33	290.67	202.67	207.00	301.00	131.33	187.33	7	230.48	69.8%	65.27	205.15	330
Hexachloroethane	330.00	330	162.67	216.33	17.67	39.67	51.00	28.00	22.67	7	76.86	23.3%	79.26	249.12	330
Nitrobenzene	330.00	330	245.33	266.33	111.33	142.33	191.67	94.67	93.67	7	163.62	49.6%	71.70	225.37	330
Isophorone	330.00	330	271.00	262.00	199.00	199.33	270.00	138.67	201.33	7	220.19	66.7%	49.49	155.55	330
2-Nitrophenol	330.00	330	216.00	220.00	111.67	141.00	201.67	90.67	103.33	7	154.90	46.9%	56.28	176.89	330
2,4-Dimethylphenol	330.00	330	233.00	206.33	82.00	150.00	220.67	112.33	124.33	7	161.24	48.9%	58.98	185.38	330
Benzoic acid	330.00	330	172.00	142.67	120.00	128.33	150.33	34.33	118.33	7	123.71	37.5%	43.69	137.31	330
bis(2-Choloroethoxy)methane	330.00	330	269.00	273.33	171.33	186.33	252.33	119.33	158.00	7	204.24	61.9%	60.60	190.45	330
2,4-Dichlorophenol	330.00	330	237.67	234.00	171.33	178.33	238.00	115.67	162.67	7	191.10	57.9%	47.01	147.76	330
1,2,4-Trichlorobenzene	330.00	330	439.00	377.00	285.33	211.00	316.00	140.00	220.67	7	284.14	86.1%	103.10	324.04	330
Naphthalene	330.00	330	241.00	254.67	115.33	150.67	199.00	100.00	110.00	7	167.24	50.7%	64.37	202.31	330
4-Chloroaniline	330.00	330	17.00	90.33	51.33	32.00	139.00	47.33	46.00	7	60.43	18.3%	41.28	129.74	330
Hexachlorobutadiene	330.00	330	212.67	227.00	70.67	106.00	139.67	66.67	60.00	7	126.10	38.2%	69.81	219.40	330
4-Chloro-3-methylphenol	330.00	330	282.00	264.00	220.00	208.67	270.33	166.67	218.33	7	232.86	70.6%	41.09	129.16	330
2-Methylnaphthalene	330.00	330	237.00	247.00	152.67	173.67	231.67	114.67	155.33	7	187.43	56.8%	51.13	160.70	330
Hexachlorocyclopentadiene	330.00	330	458.67	452.00	400.33	422.33	484.33	357.00	389.67	7	423.48	128.3%	44.45	139.71	330
2,4,6-Trichlorophenol	330.00	330	243.00	249.00	209.67	196.67	263.00	138.33	203.00	7	214.67	65.1%	42.09	132.29	330
2,4,5-Trichlorophenol	330.00	330	248.67	259.33	214.33	199.33	269.67	150.00	214.00	7	222.19	67.3%	41.23	129.57	330
2-Chloronaphthalene	330.00	330	247.00	257.67	183.33	190.00	262.67	131.33	183.00	7	207.86	63.0%	49.01	154.04	330
2-Nitroaniline	330.00	330	282.00	285.00	213.33	214.67	285.33	173.00	228.00	7	240.19	72.8%	44.40	139.56	330
Dimethylphthalate	330.00	330	271.00	276.33	246.33	221.33	293.33	190.67	239.67	7	248.38	75.3%	35.25	110.80	330
Acenaphthylene	330.00	330	252.67	267.33	203.67	209.33	277.33	156.00	207.00	7	224.76	68.1%	42.98	135.07	330
2,6-Dinitrotoluene	330.00	330	265.00	267.33	231.00	222.00	296.00	183.00	237.00	7	243.05	73.7%	36.76	115.52	330
3-Nitoraniline	330.00	330	226.00	368.00	201.00	288.00	376.33	337.67	244.67	7	291.67	88.4%	70.55	221.75	330
Acenaphthene	330.00	330	254.00	259.00	206.00	197.33	266.33	146.33	206.67	7	219.38	66.5%	43.08	135.40	330
2,4-Dinitrophenol	330.00	330	97.33	85.00	85.33	66.67	97.00	39.33	75.33	7	78.00	23.6%	20.28	63.75	330
4-Nitrophenol	330.00	330	222.33	227.67	160.00	121.67	184.67	118.00	153.33	7	169.67	51.4%	44.15	138.77	330
Dibenzofuran	330.00	330	257.67	262.00	219.00	208.00	281.00	161.00	213.33	7	228.86	69.4%	40.88	128.47	330
2,4-Dinitrotoluene	330.00	330	263.33	277.33	252.67	217.67	288.00	213.67	239.33	7	250.29	75.8%	28.45	89.40	330
Diethylphthalate	330.00	330	275.33	292.67	251.33	229.33	303.00	218.00	247.33	7	259.57	78.7%	31.86	100.13	330
4-Chlorophenyl phenylether	330.00	330	259.33	266.33	226.67	208.00	286.33	175.33	222.00	7	234.86	71.2%	38.16	119.93	330
Fluorene	330.00	330	271.67	282.67	243.67	226.00	299.67	187.00	236.33	7	249.57	75.6%	38.24	120.19	330
4-Nitroaniline	330.00	330	171.00	228.33	124.33	166.00	197.00	176.33	153.00	7	173.71	52.6%	32.83	103.18	330
1,2-Diphenylhydrazine	330.00	330	307.67	303.67	252.67	248.00	313.67	209.33	250.00	7	269.29	81.6%	39.41	123.88	330
Benzidine**	1650.00	1650	1861.00	2081.67	1839.33	2074.00	2323.67	2021.00	2110.33	7	2044.43	123.9%	163.73	514.59	1650
4,6-Dinitro-2-methylphenol	330.00	330	132.33	144.33	129.00	117.33	175.00	103.33	127.67	7	132.71	40.2%	22.59	71.01	330
N-Nitrosodiphenylamine	330.00	330	406.67	431.33	322.67	349.33	435.67	301.67	344.67	7	370.29	112.2%	53.85	169.24	330
4-Bromophenyl phenylether	330.00	330	249.67	256.00	214.67	217.00	270.67	173.33	220.00	7	228.76	69.3%	32.71	102.82	330
Hexachlorobenzene	330.00	330	265.33	277.67	234.33	239.00	291.67	198.00	239.33	7	249.33	75.6%	31.41	98.71	330
Pentachlorophenol	330.00	330	170.33	174.33	175.67	149.33	194.00	131.33	173.67	7	166.95	50.6%	20.40	64.13	330

MDL 2007 SVOC Solids

Analytical Method: SW846 8270C

Extraction Method: SW846 3550B

			Replicate Number												
			1	2	3	4	5	6	7						
	Extract Date		3/19/2007	3/19/2007	3/19/2007	3/21/2007	3/26/2007	3/26/2007	4/4/2007						
	Analysis Date		4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/11/2007	4/12/2007						Reporting
	Spike Conc	Est. Limit									Samples	Mean	Average	Deviation	MDL	Limit
Compound	(ug/kg)	(ug/kg)									(N)	(ug/kg)	%Rec.	(s)	(ug/kg)	(ug/kg)
Phenanthrene	330.00	330	277.33	288.67	237.67	235.67	294.33	210.67	241.33		7	255.10	77.3%	31.64	99.44	330
Anthracene	330.00	330	258.00	266.00	223.67	217.67	281.00	199.67	225.00		7	238.71	72.3%	29.68	93.30	330
Carbazole	330.00	330	330.00	285.67	276.67	277.33	352.00	270.33	289.33		7	297.33	90.1%	31.12	97.82	330
Di-n-butylphthalate	330.00	330	278.00	283.67	246.00	231.00	287.00	224.67	239.33		7	255.67	77.5%	26.44	83.10	330
Fluoranthene	330.00	330	257.67	273.33	241.00	222.00	279.00	218.67	230.67		7	246.05	74.6%	24.34	76.51	330
Pyrene	330.00	330	286.67	294.33	245.00	228.00	293.00	229.33	246.67		7	260.43	78.9%	29.85	93.81	330
Butylbenzylphthalate	330.00	330	277.00	280.33	230.33	215.33	283.00	217.00	236.67		7	248.52	75.3%	30.49	95.83	330
3,3'-Dichlorobenzidine**	1650.00	1650	921.67	1047.00	919.00	1055.00	1126.33	1077.67	1101.00		7	1035.38	62.8%	83.03	260.96	1650
Benzo(a)anthracene	330.00	330	267.00	276.67	228.00	216.67	281.00	215.67	226.00		7	244.43	74.1%	29.14	91.58	330
Chrysene	330.00	330	268.33	280.00	238.67	222.00	284.33	232.67	240.67		7	252.38	76.5%	24.75	77.79	330
bis(2-Ethylhexyl)phthalate	330.00	330	291.67	284.33	301.33	336.00	341.33	276.33	327.00		7	308.29	93.4%	26.23	82.46	330
Di-n-octylphthalate	330.00	330	307.67	289.67	257.67	222.33	289.67	217.67	236.33		7	260.14	78.8%	36.08	113.39	330
Benzo(b)fluoranthene	330.00	330	299.00	292.67	267.67	223.00	317.00	237.67	251.67		7	269.81	81.8%	34.54	108.57	330
Benzo(k)fluoranthene	330.00	330	318.33	321.67	291.33	254.67	325.67	255.33	276.00		7	291.86	88.4%	30.83	96.89	330
Benzo(a)pyrene	330.00	330	268.00	274.67	221.33	209.00	269.33	214.67	218.67		7	239.38	72.5%	29.58	92.97	330
Indeno(1,2,3-cd)pyrene	330.00	330	276.33	271.00	256.00	226.33	292.00	229.00	242.00		7	256.10	77.6%	24.96	78.46	330
Dibenz(a,h)anthracene	330.00	330	265.67	259.00	253.33	218.67	290.33	222.67	236.67		7	249.48	75.6%	25.39	79.81	330
Benzo(g,h,i)perylene	330.00	330	304.33	307.67	300.00	258.67	335.33	260.33	278.33		7	292.10	88.5%	27.81	87.40	330

**Analytes extracted 4/26/07; analyzed on 4/28/07

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/OA/1500

PCBs by Gas Chromatography (GC)

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 2

SOP Effective Date: 6/12/07

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Approved By_____
Date _____

1.0 Methods

1.1 This SOP is applicable to the following methods: SW846 (8082), and 40 CFR Method 608.

2.0 Applicable Matrices

2.1 This method applies to extracts from a wide variety of environmental matrices, including ground water, aqueous sludges, soils, sediments, acid and caustic liquors, oils, solvents, polymers, wipe and waste materials.

3.0 Detection Limit

3.1 Detection limits vary per compound Detection limit studies are run annually to determine performance Current detection limit performance and limits can be found with the QA/QC Officer.

4.0 Scope and Application

4.1 This method is used to determine the concentrations of PCBs as Aroclors in extracts from various matrices. Open-tubular capillary columns are employed with electron capture detectors (ECD) The following list describes applicable PCBs:

Compound	CAS Number
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

4.2 Aroclors are multi-component mixtures; a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis.

4.3 Compound identification is confirmed by use of a second column

4.4 MDL studies are performed on an annual basis; see the latest QA/QC documentation detailing the latest MDL study results and reporting limits.

5.0 Summary

5.1 A measured volume or weight is extracted using a matrix appropriate extraction technique

5.2 Extracts may be subjected to sulfuric acid/potassium permanganate cleanup. This cleanup will destroy many single component organochlorine or organosphosphorous pesticides. This may not be desirable if analyzing Method 608 with Pesticides.

5.3 After extraction and clean-up, the extract is injected into a GC with an ECD detector

5.4 The chromatographic data is interpreted to determine the seven Aroclors from 4.1.

6.0 Responsibilities

6.1 Analyst: Personnel experienced in the use of gas chromatograph/electron capture detectors, and skilled in the interpretation of chromatograms and their use as a quantitative tool. The analyst has demonstrated the ability to generate acceptable results using this SOP or is under the direct supervision of analysts experienced these techniques. Analyst must perform an initial demonstration of capability prior to reporting results.

7.0 Definitions

7.1 Surrogate: A pure analyte, which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction and is measured with the same procedures used to measure other sample components. The purpose of the surrogate is to monitor method performance with each sample

7.2 LCS: Laboratory control sample, an aliquot of reagent water to which a known amount of method analytes are added. It is analyzed like a sample and used to determine whether the method is in control. LCS samples are produced using the secondary source standard

7.3 SSS: Secondary source standard, solution purchased from a separate supplier than the primary source standards or from the same supplier with a different lot number Used to validate a new calibration curve

7.4 MS/MSD: Matrix spike and matrix spike duplicate, an aliquot of a selected sample is spiked with a known amount of the method analytes It is analyzed like a sample and used to determine the presence of matrix interferences. These samples are produced using the secondary source standard.

7.5 Method Blank: An aliquot of reagent water that is treated as a sample and used to determine if method analytes or other interferences are present.

7.6 GC: Gas chromatograph

7.7 GC-ECD: Gas Chromatograph/Electron Capture Detector

7.8 CCC: Continuing Calibration Check standard Run with each analytical batch: used to verify that the system still falls within calibration.

8.0 Interferences

8.1 Interferences can vary considerably from sample to sample and matrix to matrix. Cleanup techniques can be used to improve quantitation The following sources can cause interferences:

8.1.1 Contaminated solvents or reagents

8.1.2 Contaminated carrier gas, parts, column or detector surfaces.

8.1.3 Compounds extracted to which the detector will respond

8.2 Interferences by phthalate esters can pose problems in PCB determination This can be avoided by minimizing contact with any plastic materials and checking all solvents and reagents for phthalate contamination Sulfuric acid/permanganate clean-up can remove phthalates.

8.3 Keep glassware clean:

8.3.1 Clean all glassware as soon as possible after use by rinsing with the last solvent used. Then clean thoroughly with reagent water. Oven-dry glassware.

8.3.1.1 Drying glassware that has been exposed to high PCBs may contaminate other glassware. Use care when drying

8.4 Elemental sulfur is readily extracted from soil samples and can cause interference. Clean-up procedures can be used to eliminate this interference.

9.0 Environmental Health and Safety

9.1 This method uses standards and reagents that are extremely hazardous. All sample and standard preparation should be performed in a fume hood. The wearing of lab coat, safety glasses and gloves is highly recommended to avoid exposure to hazardous agents.

9.2 Consult the FIRSTECHNOLOGY Chemical Hygiene Plan and individual MSDS forms to determine the health hazards of specific compounds

10.0 Equipment and Supplies

10.1 GC System

10.1.1 Gas Chromatograph: 5890 Series II Plus GC from Hewlett-Packard (HP) equipped with two columns and two ECDs, with Chem Station software for data analysis.

10.1.2 Two columns are mounted in the GC, with each column connected to a separate injector and a separate ECD.

10.1.3 Column #1 (Front Column) - RTX-CLP Pesticides - 30 m X 0.32 mm x 0.25 µm film thickness fused silica capillary column

10.1.4 Column #2 (Back Column) - RTX-CLP Pesticides 2 - 30 m X 0.32 mm x 0.25 µm film thickness fused silica capillary

10.1.5 Autosampler - 6890 from HP

10.2 Autosampler vials (2 mL)

11.0 Reagents

11.1 Reagent or pesticide grade solvents for extraction and standard preparation

11.2 Organic-free reagent-grade water

11.3 All stock standard solutions are purchased as certified solutions from various vendors and stored at 4°C as recommended by the manufacturer. Stock standards are replaced after one year. Standards and reagents may be purchased from any vendor with the exception of calibration standards and calibration check standards, which must be obtained from two different sources.

11.4 PCB calibration standards are prepared by diluting a stock calibration standard mixture of Aroclor-1016 and Aroclor-1260 to 5 to 8 different concentration levels. Single concentration standards of each of the remaining Aroclors also are analyzed with each calibration. Concentrations of calibrations standards are listed in Table 1. Calibration standards are prepared whenever a new calibration is performed

Table I: PCB Calibration Standard Concentrations

Standard Concentration (ug/mL)	Preparation Volumes
0.2	2 uL (100 ug/mL std) to 1 mL Hexane
0.5	5 uL (100 ug/mL std) to 1 mL Hexane
1.0	10 uL (100 ug/mL std) to 1 mL Hexane
2.0	20 uL (100 ug/mL std) to 1 mL Hexane
3.0	30 uL (100 ug/mL std) to 1 mL Hexane
4.0	40 uL (100 ug/mL std) to 1 mL Hexane
5.0	50 uL (100 ug/mL std) to 1 mL Hexane
10.0	100 uL (100 ug/mL std) to 1 mL Hexane

11.5 PCB calibration check standard is prepared by diluting a stock calibration check standard mixture of Aroclor-1016 and Aroclor-1260 to 2.0 µg/mL; it stored at 4°C and prepared monthly

11.6 Pesticide calibration standards are prepared by diluting a stock calibration standard to 5 to 8 different concentration levels A set of calibration standards (5 to 8 different concentration levels) for pesticides not present in the primary standard mixture (e.g., Chlordane, Toxaphene, etc.) also is prepared and analyzed with each calibration Calibrations for other pesticides are performed on an as needed basis Concentrations of calibrations standards are listed in Table I. Calibration standards are prepared whenever a new calibration is performed.

11.7 Pesticide calibration check standard is prepared by diluting a stock calibration check standard mixture to 2.0 ug/mL (1.0 ug/mL for Toxaphene and 4 0 ug/mL for Chlordane); it is stored at 4°C and prepared monthly.

11.8 Surrogate stock standard, a mixture of 2,4,5,6-Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCBP), is prepared at 50 µg/mL for each surrogate

12.0 Sample Collection, Preservation and Handling

12.1 Samples must be collected in pre-cleaned glass containers. Water samples will be collected in 1000 ml amber bottles, and soil in soil jars Both are kept at 4°C ±2°C

12.2 Water samples must be extracted within 7 days of sampling, soils within 14 days.

12.3 Store sample extracts at -10°C, protected from light, in sealed vials (e g , screw-cap vials or crimp-capped vials) equipped with un-pierced PTFE-lined septa. Extracts must be analyzed within 40 days.

13.0 Quality Control

13.1 A preparation batch of samples is defined as a group of up to twenty field samples of similar matrix type that have been prepared at the same time or time sequence with the same lots of reagents for the same analysis. In addition to the twenty samples, each preparatory batch will contain at a minimum, a method blank, a laboratory control sample, a matrix spike, a matrix spike duplicate and a matrix duplicate. An analytical, or instrumental batch is defined as samples that are analyzed together within the same time period or in continuous sequential time periods

13.2 An initial demonstration of proficiency must be performed for each instrument and analyst by extracting and analyzing four replicates of a calibration standard at a concentration of 20-100 µg/mL. This step is performed by all analysts at least once per year and for new analyst training.

13.3 All samples in an extraction batch for PCBs (QC and client samples) must be analyzed, although they do not need to be analyzed in the same analytical batch or run.

13.4 The method blank analysis is reviewed for presence of target analytes. If any are detected, all samples in the extraction batch associated with that method blank are reviewed for that analyte. Laboratory contamination is considered a possibility if the client sample concentration of an analyte detected in the method blank is less than 5 times the level in the method blank.

13.5 An analytical run consists of a calibration check standard and any samples run within a 12-hr period. The 12-hr period begins at the injection time of the tuning standard. QC batch and individual sample criteria that are evaluated include: percent recoveries for surrogates, LCS, MS and MSD; and relative standard deviation (RSD) for MS vs. MSD. These statistical criteria are compared with laboratory-derived and/or method-specified control limits.

14.0 Procedure

14.1 The GC is set up per conditions described in Table II. All injections are 1 µL, administered through the autosampler.

Table II: Operating Conditions

Column: SPB-5
30m X 0.53 lm
5.0 lm film thickness
Head pressure: 4 psi

Confirmatory column: SPB-608
30m X 0.53 lm
5.0 lm film thickness
Head pressure: 3 psi

Initial temp: 150°C
Hold: 4 min.
Rate: 4°/min
Final temp: 290°C
Final time: 21 min.

14.2 <u>PCB Initial Calibration</u>:

14.2.1 An initial calibration is performed when an instrument is first set up, when a column or source is changed, or when any major maintenance is performed on the instrument. It also is performed at a minimum of once per year or whenever continuing calibration check criteria, relative to the initial calibration, can not be achieved.

14.2.2 A set of 5 to 8 calibration standards, containing Aroclor-1016/1260 as well as surrogates (TCX and DCBP) at concentrations listed in Table I is prepared. A single concentration calibration standard (2.0 µg/mL), also containing surrogates TCX and DCBP at 0.5 µg/mL is prepared for each of the other Aroclors. Each standard (0.5 mL) is placed into a 2-mL autosampler vial and 1 µL is injected for analysis.

14.2.3 Aroclor responses for the Aroclors and surrogates are determined using an external standard technique. The elution profile for each Aroclor or surrogate is examined to select peaks (5 peaks each for Aroclor-1016 and -1260, 3-5 peaks each for the other

Aroclors) that together define that Aroclor; surrogates have one peak each. A calibration factor (CF) is determined for each peak, as follows:

$$CF = \frac{\text{Peak Height in the Standard}}{\text{Concentration of Standard } (\mu g/mL)}$$

14.2.4 For each Aroclor-1016, Aroclor-1260, TCX and DCBP peak, statistical values are calculated for the CFs: mean, standard deviation, and relative standard deviation (RSD). A calibration is acceptable for an individual compound if the RSDs for all peaks of that compound are less than or equal to 20%

14.2.5 For the other Aroclors, the CF is calculated for each peak that defines that Aroclor, using the single concentration analysis

14.2.6 Retention time windows for each compound peak are determined using a minimum of three runs over a 72 hour period for Aroclor-1016, Aroclor-1260, TCX and DCBP. The retention window will be calculated by taking plus and minus three times the standard deviation of the retention times For the other Aroclors (analyzed at 2.0 µg/mL), the retention time of each peak that defines that Aroclor is noted.

14.2.7 All above criteria are determined for the primary analysis column (usually, but not necessarily always, Column #1 - the Front Column.) The secondary column (Column #2 - the Back Column) is used for confirmation of compound identification, and therefore only the peak retention times are evaluated from this column.

14.2.8 If the above calibration criteria are not met, the source of the problem must be identified, corrected, and the initial calibration repeated, until criteria are met, before proceeding to the next step of this procedure

14.3 PCB Daily Calibration or Continuing Calibration Verification:

14.3.1 A PCB continuing calibration check standard (2.0 µg/mL each of Aroclor 1016 and Aroclor-1260) is prepared. This check standard (1.0 mL) is placed into a 2-mL autosampler vial. Surrogates (TCX and DCBP, 25 µL of 100 µg/mL) are added to the vial and the mixture (1 µL) is injected for analysis

14.3.2 For an acceptable calibration check, the percent difference of the calibration factors (CF) between the CCC and initial calibration must not exceed 15%.

14.3.3 If the above continuing calibration criteria are not met, the source of the problem must be identified, corrected, and the continuing calibration repeated, until criteria are met, before proceeding to the next step of this procedure

14.4 GC Analysis of Sample:

14.4.1 A sample extract (1 mL) is received, following preparation according to the procedure appropriate for the sample matrix, in a 2-mL autosampler vial 1 µL is injected for analysis

15.0 Calculations

15.1 Calculation of calibration factors:

$$CF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate
A_{is}= Peak area (or height) of the internal standard
C_s = Concentration of the analyte or surrogate, in ug/L.
C_{is} = Concentration of the analyte or internal standard, in ug/L.

15.2 Average value

$$\overline{X} = \frac{1}{n}\sum X$$

where:

X = value to be averaged
n = the number of values
$\overline{X}$ = the average

15.3 Calculation of % Difference

$$\%\ \text{Difference} = \frac{CF_v - CF}{\overline{CF}} \times 100$$

where:

CF_v = ca;ibration factor from verified standard
CF = Mean calibration factor from the initial calibration.

15.4 Sample concentration for liquid (ug/L) and soil samples (ug/Kg)

$$\text{Concentration} = \frac{\left[\dfrac{A_x \times C_{is}}{\overline{CF} \times A_{is}}\right]}{S} \times D$$

where:

A_x = Response for the analyte in the sample, in area counts
D = Dilution factor
$\overline{CF}$ = Mean CF value from the initial calibration, in counts per ng
C_{is} = Concentration of internal standard in ug/mL
A_{is} = Area count of associated internal standard
S = Weight or volume of sample

15.5 Calculation of Recovery

Surrogates:

$$\%\text{Recovery} = \frac{C_m}{C_s} \times 100$$

where:

C_m = Measured concentration of the surrogate
C_s = Nominal (or theoretical) Concentration of the surrogate

MS/MSD and LCS analyses:

$$\%\text{Recovery} = \frac{C_s - C_u}{C_n} \times 100$$

where:

C_s = Measured concentration of the spiked sample aliquot
C_u = Measured concentration of the un-spiked sample aliquot (use 0 for LCS analyses)
C_n = Nominal (or theoretical) concentration of the spiked sample aliquot

15.6 Calculation of Precision

$$\text{RPD} = \frac{(C_1 - C_2)}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where:

C_1 = Measured concentration of the first sample
C_2 = Measured concentration of the second sample

15.7 Best-fit line (linear regression)

15.7.1 Step 1; minimize the sum of the squares

$$S_{xx} = \sum \left(x_i - \bar{x}\right)^2$$

where:

S_{xx} = sum of squares for concentration of standard solution
X_i = individual data points for concentration
$\overline{X}$ = average value for concentrations

$$S_{yy} = \sum \left(y_i - \bar{y}\right)^2$$

where:

S_{yy} = sum of squares for area of a peak
Y_i = individual data points for area of peak
$\overline{Y}$ = average value for area of peaks, see 10.2

$$S_{xy} = \sum \left(X_i - \overline{X}\right)\left(Y_i - \overline{Y}\right)$$

where:

S_{xy} = sum of squares for concentration of standard solution
X_i = individual data points for concentration
$\overline{X}$ = average value for concentrations
Y_i = individual data points for area of peak
$\overline{Y}$ = average value for area of peak

15.7.2 Step 2; calculate the slope and intercept

$$m_{LS} = \frac{S_{xx}}{S_{yy}}$$

where:

S_{xx} = sum of squares for concentration of standard solution
S_{yy} = sum of squares for area of a peak
m_{LS} = least-squares slope

$$b_{LS} = \overline{Y} - m_{LS}\overline{X}$$

where:

$\overline{Y}$ = average value for area of peak
$\overline{X}$ = average value for concentration
m_{LS} = least-squares slope
b_{LS} = least-squares intercept

15.7.3 Step 3; calculate the best-fit line

$$Y = m_{LS}X + b_{LS}$$

where:

Y = calculated peak areas
m_{LS} = least-squares slope
b_{LS} = least-squares intercept
X = concentrations

16.0 Data Validation

16.1 Qualitative Analysis: The Chem Station software is used to qualitatively evaluate the sample profile (chromatographic and mass specta) relative to the initial calibration curve valid for the analytical run. Target compounds peaks identified by the Chem Station software are examined. The relative retention time of each compound must be within 0.06 min of its retention time in the initial calibration. The relative intensities of the characteristic mass ions for each compound must agree within 30% of the relative intensities of these ions for the same compound in the mid-level standard of the initial calibration set. The Chem Station evaluation is examined and verified for each sample. If necessary, the compound identification is expanded to include Chem Station library search compounds. If this search is needed, it it performed manually.

16.2 **Qualitative Analysis:**

2007 MDL Study Pesticide, Herbicide, PCB Liquid

Compound	Est. Limit (ug/L)	Spike Conc. (ug/L)	Prep Date		Replicate Number 1	2	3	4	5	6	7	Samples (N)	Mean Rec. (ug/L)	Average %Rec	Deviation (s)	Calc. MDL ug/L	Reporting Limit ug/L
Pesticides																	
Analysis Method: SW846-8081A																	
Extraction Method: SW846-3510C				**Anal. Date**	**2/19/07**	**2/19/07**	**2/19/07**	**2/20/07**	**2/20/07**	**2/20/07**	**2/21/07**						
alpha-BHC - 1.0	0.25	0.10	2/5-2/7/07		0.1048	0.1095	0.1060	0.1040	0.1046	0.1051	0.0996	7	0.1048	104.8%	0.0029	0.0092	0.25
gamma-BHC (Lindane) - 1.0	0.25	0.10	2/5-2/7/07		0.1049	0.1113	0.1069	0.1051	0.1061	0.1063	0.1007	7	0.1059	105.9%	0.0031	0.0099	0.25
beta-BHC - 1.0	0.25	0.10	2/5-2/7/07		0.1072	0.1156	0.1105	0.1060	0.1093	0.1093	0.1017	7	0.1085	108.5%	0.0043	0.0134	0.25
delta-BHC - 1.0	0.25	0.10	2/5-2/7/07		0.1041	0.1107	0.1065	0.1025	0.1035	0.1039	0.0965	7	0.1040	104.0%	0.0043	0.0135	0.25
Heptachlor - 1.0	0.25	0.10	2/5-2/7/07		0.1100	0.1143	0.1094	0.1090	0.1086	0.1099	0.1070	7	0.1097	109.7%	0.0022	0.0071	0.25
Aldrin - 1.0	0.25	0.10	2/5-2/7/07		0.1060	0.1107	0.1067	0.1052	0.1058	0.1066	0.1023	7	0.1062	106.2%	0.0025	0.0078	0.25
Heptachlor epoxide - 1.0	0.25	0.10	2/5-2/7/07		0.1178	0.1230	0.1195	0.1162	0.1191	0.1196	0.1142	7	0.1185	118.5%	0.0028	0.0088	0.25
DDE - 1.0	0.25	0.10	2/5-2/7/07		0.1044	0.1093	0.1053	0.1034	0.1046	0.1061	0.1028	7	0.1051	105.1%	0.0021	0.0067	0.25
Endosulfan I - 1.0	0.25	0.10	2/5-2/7/07		0.1057	0.1130	0.1071	0.1052	0.1066	0.1126	0.1043	7	0.1078	107.8%	0.0035	0.0111	0.25
Dieldrin - 1.0	0.25	0.10	2/5-2/7/07		0.1094	0.1157	0.1120	0.1096	0.1133	0.1145	0.1102	7	0.1121	112.1%	0.0025	0.0078	0.25
Endrin - 1.0	0.25	0.10	2/5-2/7/07		0.1083	0.1135	0.1106	0.1109	0.1124	0.1135	0.1123	7	0.1116	111.6%	0.0019	0.0058	0.25
DDD - 1.0	0.25	0.10	2/5-2/7/07		0.1042	0.1005	0.1071	0.1097	0.1120	0.1166	0.1048	7	0.1078	107.8%	0.0054	0.0169	0.25
Endosulfan II - 1.0	0.25	0.10	2/5-2/7/07		0.1066	0.1138	0.1101	0.0984	0.1106	0.1128	0.1092	7	0.1088	108.8%	0.0051	0.0162	0.25
DDT - 1.0	1.0	0.10	2/5-2/7/07		0.1076	0.1149	0.1113	0.1141	0.1175	0.1187	0.1194	7	0.1148	114.8%	0.0042	0.0133	1.0
Endrin Aldehyde - 1.0	0.25	0.10	2/5-2/7/07		0.0992	0.1048	0.1018	0.1020	0.1040	0.1069	0.1052	7	0.1034	103.4%	0.0026	0.0081	0.25
Methoxychlor - 1.0	1.0	0.10	2/5-2/7/07		0.0996	0.1032	0.1456	0.1098	0.1056	0.1080	0.1101	7	0.1117	111.7%	0.0154	0.0484	1.0
Endosulfan sulfate - 1.0	1.0	0.10	2/5-2/7/07		0.1018	0.1119	0.1089	0.1078	0.1111	0.1119	0.1120	7	0.1093	109.3%	0.0037	0.0117	1.0
Endrin ketone - 1.0	0.25	0.10	2/5-2/7/07		0.1072	0.1170	0.1456	0.1098	0.1169	0.1080	0.1202	7	0.1178	117.8%	0.0132	0.0416	0.25
Chlordane	0.50	0.10	2/5-2/7/07		0.1055	0.1110	0.1070	0.1043	0.1061	0.1070	0.1030	7	0.1063	106.3%	0.0025	0.0080	0.50
Toxaphene	5.0	0.10	2/5-2/7/07		0.104	0.112	0.106	0.104	0.105	0.111	0.102	7	0.1063	106.3%	0.0035	0.0111	5.0
Herbicides																	
Analysis Method: SW846-8151A																	
Extraction Method: SW846-8151A				**Anal.Date**	**4/20/07**	**4/20/07**	**4/20/07**	**4/23/07**	**4/23/07**	**4/23/07**	**4/23/07**						
Dicamba - 1.0	0.50	0.01	4/18/2007		0.0093	0.0091	0.0089	0.0083	0.0070	0.0083	0.0084	7	0.0085	84.7%	0.0008	0.0024	0.50
Dichlorprop - 10	5.0	0.01	4/18/2007		0.0099	0.0099	0.0096	0.0090	0.0079	0.0090	0.0091	7	0.0092	92.0%	0.0007	0.0022	5.0
2,4-D - 10	5.0	0.01	4/18/2007		0.0093	0.0093	0.0091	0.0085	0.0074	0.0084	0.0084	7	0.0086	86.3%	0.0007	0.0021	5.0
2,4,5-TP (Silvex) - 1.0	0.50	0.01	4/18/2007		0.0095	0.0093	0.0093	0.0083	0.0076	0.0084	0.0085	7	0.0087	87.0%	0.0007	0.0022	0.50
2,4,5-T - 1.0	0.50	0.01	4/18/2007		0.0094	0.0094	0.0090	0.0082	0.0077	0.0082	0.0082	7	0.0086	85.9%	0.0007	0.0021	0.50
2,4-DB - 10	5.0	0.01	4/18/2007		0.0096	0.0105	0.0090	0.0098	0.0092	0.0095	0.0096	7	0.0096	96.0%	0.0005	0.0015	5.0
Dinoseb - 5.0	2.5	0.01	4/18/2007		0.0023	0.0016	0.0022	0.0018	0.0014	0.0015	0.0015	7	0.0018	17.6%	0.0004	0.0012	2.5
PCB																	
Analysis Method: SW846-8082																	
Extraction Method: SW846-3510C				**Anal. Date**													
PCB-1016	0.10	0.2	2/5-2/7/07	2/7/2007	0.2700	0.2700	0.2600	0.2700	0.2700	0.2700	0.2600	7	0.2671	133.6%	0.0049	0.0153	0.10
PCB-1221	0.10	0.005	4/18/2007	4/19/2007	0.0047	0.0050	0.0047	0.0049	0.0053	0.0052	0.0052	7	0.0050	100.0%	0.0002	0.0008	0.10
PCB-1232	0.10	0.002	4/27/2007	4/28/2007	0.0021	0.0021	0.0021	0.0021	0.0021	0.0021	0.0022	7	0.0021	105.7%	0.0000	0.0001	0.10
PCB-1242	0.10	0.002	4/24/2007	4/27/2007	0.0034	0.0027	0.0027	0.0028	0.0028	0.0026	0.0028	7	0.0028	141.4%	0.0003	0.0008	0.10
PCB-1248	0.10	0.002	4/27/2007	4/29/2007	0.0021	0.0021	0.0020	0.0021	0.0021	0.0020	0.0019	7	0.0020	102.1%	0.0001	0.0003	0.10
PCB-1254	0.10	0.002	4/26/2007	4/28/2007	0.0024	0.0024	0.0024	0.0024	0.0023	0.0020	0.0018	7	0.0022	112.1%	0.0002	0.0008	0.10
PCB-1260	0.10	0.5	2/5-2/7/07	2/7/2007	0.3400	0.3400	0.3500	0.3500	0.3500	0.3600	0.3600	7	0.3500	70.0%	0.0082	0.0257	0.10

2007 MDL Study Pesticide, PCB Solid

Compound	Est. Limit (ug/kg)	Spike Conc. (ug/kg)	Extract. Date	Analysis Date	Replicate Number 1	2	3	4	5	6	7	Samples (N)	Mean Rec. (ug/kg)	Average % Rec.	Deviation (s)	Calc. MDL ug/Kg	Reporting Limit ug/Kg
Pesticides																	
Analysis Method: SW846-8081A																	
Extraction Method: SW846-3550B					2/19/07	2/19/07	2/19/07	2/20/07	2/20/07	2/22/07	2/22/07						
alpha-BHC	2.00	5.00	2/5-2/7/07		5.3052	5.5762	5.2544	5.1140	5.2093	4.9250	5.1472	7	5.2188	104.4%	0.1848	0.5807	2.00
gamma-BHC (Lindane)	2.00	5.00	2/5-2/7/07		5.3503	5.5762	5.4497	5.1710	5.3100	5.0113	5.2061	7	5.2964	105.9%	0.1732	0.5444	2.00
beta-BHC	2.00	5.00	2/5-2/7/07		5.4220	5.7621	5.6582	4.8292	5.4022	5.0694	5.4053	7	5.3641	107.3%	0.2987	0.9387	2.00
delta-BHC	2.00	5.00	2/5-2/7/07		5.1608	5.5662	5.4531	4.9730	5.1967	4.8115	5.1528	7	5.1877	103.8%	0.2400	0.7545	2.00
Heptachlor	2.00	5.00	2/5-2/7/07		5.5082	5.9364	5.9087	5.4340	5.5103	5.3097	5.5175	7	5.5893	111.8%	0.2213	0.6955	2.00
Aldrin	2.00	5.00	2/5-2/7/07		5.3661	5.6206	5.3130	5.1652	5.2878	5.0980	5.2731	7	5.3034	106.1%	0.1545	0.4856	2.00
Heptachlor epoxide	2.00	5.00	2/5-2/7/07		5.9901	6.3329	5.5475	5.7585	5.9541	5.7558	5.9954	7	5.9049	118.1%	0.2307	0.7250	2.00
DDE	2.00	5.00	2/5-2/7/07		5.2488	5.5355	5.3988	5.0996	5.2741	5.3041	5.4404	7	5.3288	106.6%	0.1323	0.4160	2.00
Endosulfan I	2.00	5.00	2/5-2/7/07		5.4144	5.6481	5.6510	5.1824	5.3948	5.2412	5.4577	7	5.4271	108.5%	0.1671	0.5253	2.00
Dieldrin	2.00	5.00	2/5-2/7/07		5.6090	5.9382	5.7627	5.4547	5.6797	5.6275	5.8729	7	5.7064	114.1%	0.1533	0.4817	2.00
Endrin	2.00	5.00	2/5-2/7/07		5.3780	5.8538	6.0889	5.3926	5.6794	5.8386	6.0728	7	5.7577	115.2%	0.2695	0.8470	2.00
DDD	2.00	5.00	2/5-2/7/07		5.4603	5.8787	5.7170	5.4904	5.7193	5.9271	6.1636	7	5.7652	115.3%	0.2301	0.7233	2.00
Endosulfan II	2.00	5.00	2/5-2/7/07		5.3796	5.7680	5.6933	5.3626	5.6371	5.6981	5.9637	7	5.6432	112.9%	0.1972	0.6196	2.00
DDT	2.0	5.00	2/5-2/7/07		5.5251	5.9942	5.8012	5.6589	5.9998	6.4758	6.6910	7	6.0209	120.4%	0.3932	1.2358	2.0
Endrin Aldehyde	2.00	5.00	2/5-2/7/07		5.0326	5.3353	5.2263	4.9974	5.2107	5.4906	5.6674	7	5.2800	105.6%	0.2224	0.6990	2.00
Methoxychlor	5.0	5.00	2/5-2/7/07		5.0685	5.9174	8.6662	5.4780	5.3526	5.8861	7.5212	7	6.2700	125.4%	1.2242	3.8478	5.0
Endosulfan sulfate	2.0	5.00	2/5-2/7/07		5.2016	5.6932	5.5931	5.2599	5.5972	5.9651	6.1331	7	5.6347	112.7%	0.3145	0.9883	2.0
Endrin ketone	5.00	5.00	2/5-2/7/07		5.4863	5.9174	5.7954	5.6495	5.9509	6.3910	7.5212	7	6.1017	122.0%	0.6361	1.9993	5.00
Chlordane	2.0	5.00	2/5-2/7/07		5.33960	5.61810	5.37660	5.13940	5.32150	5.21770	5.39490	7	5.3440	106.9%	0.1400	0.43990	2.0
Toxaphene	2	5.0	2/5-2/7/07		5.3666	5.5626	5.3182	5.0838	5.3154	5.2165	5.3742	7	5.3196	106.4%	0.1365	0.4291	2
PCB																	
Analysis Method: SW846-8082																	
Extraction Method: SW846-3550B				**Anal.Date**													
PCB-1016	100	100.0	1/23-1/24/07	1/29/2007	135.0	130.0	125.0	100.0	95.0	90.0	130.0	7	115.0000	115.0%	17.7281	55.7194	100
PCB-1221	50	250.0	4/16-4/18/07	4/18/2007	275.0	270.0	270.0	275.0	250.0	270.0	270.0	7	268.5714	107.4%	7.8895	24.7968	50
PCB-1232	100	400.0	11/3/06	11/7/06	336.8	358.6	334.3	332.9	374.0	314.6	340.4	7	341.6343	85.4%	17.8100	55.9769	100
PCB-1242	50	100.0	4/2-4/4/07	4/5/2007	135.0	140.0	140.0	140.0	130.0	125.0	135.0	7	135.0000	135.0%	5.3452	16.8000	50
PCB-1248	50	200.0	11/2/06	11/6/06	168.0	164.5	172.3	165.4	161.9	157.0	154.0	7	163.3150	81.7%	5.8092	18.2584	50
PCB-1254	50	100.0	3/20-3/22/07	3/22/2007	145.0	150.0	160.0	155.0	155.0	155.0	145.0	7	152.1429	152.1%	5.2489	16.4973	50
PCB-1260	50	100.0	1/23-1/24/07	1/29/2007	135.0	135.0	130.0	135.0	140.0	140.0	155.0	7	138.5714	138.6%	7.4231	23.3307	50

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/OE/1500

General Organic Extraction Procedures

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 2

SOP Effective Date: 5/31/07

Copy No. _____
Approved By_____
Date _____

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1.0 Scope and Application

1.1 This document provides general procedures and information regarding organic extraction procedures. It details general health and safety information, standard glassware washing procedures, and common quality control procedures.

1.2 Specific information for each type of extraction is found in individual extraction SOP documents.

2.0 Definitions

2.1 **BNA** - base/neutral, acid compounds

2.2 **conc.** - concentrate or concentrated

2.3 **DI** water - deionized water

2.4 **evap. flask** - evaporating flask

2.5 **herb.** – herbicide

2.6 **KD** - Kuderna-Danish concentrating apparatus

2.7 **pest.** – pesticide

2.8 **RB flask** - round bottom flask

2.9 **sep. funnel** - separatory funnel, particular size to be used is determined by the amount of original liquid sample used

2.10 **SOP** - Standard Operating Procedure

2.11 **SONI** – Sonication Extraction

2.12 **VOC** – Volatile Organic Compound

2.13 **w.d.** - waste dilution

3.0 Environmental Health and Safety

3.1 Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

3.2 Wear proper laboratory protective equipment such as proper lab safety goggles and a lab coat. If extracting samples using organic solvents, wear solvent resistant non-latex gloves.

3.3 Vent and pour solvents under a fume hood providing sufficient ventilation

3.4 Consult the FIRSTECHNOLOGY Chemical Hygiene Plan and specific chemical MSDS sheets for health and safety information on individual chemicals

4.0 General Procedures

4.1 Glassware Cleaning-General

4.1.1 Rinse glassware with last solvent used.
4.1.2 Soak the glassware in hot water.
4.1.3 Wash with hot soap & water. Use the correct sized brush
4.1.4 Rinse with tap water.
4.1.5 Rinse with reagent-grade water.
4.1.6 Allow to drip dry on a rack.

4.2 Glassware Cleaning After PCBs

4.2.1 Rinse with solvent *last* used.
4.2.2 Soak in hot water
4.2.3 Wash with hot soap & water, brush thoroughly.
4.2.4 Rinse with hot water.
4.2.5 Soak in no-chromix solution for 1 hour.
4.2.6 Rinse with hot tap water.
4.2.7 Rinse with reagent-grade water.
4.2.8 Rinse with acetone

4.3 CLEANING OF GLASSWARE FOR HERBICIDES

4.3.1 Glassware must be washed with hot tap water and soap.
4.3.2 Rinse with 1:9 H_2SO_4.
4.3.3 Rinse with tap water.
4.3.4 inse with reagent-grade water.
4.3.5 Rinse with acetone.
4.3.6 If time permits, put in oven for approximately 1 hour.
4.3.7 After drying, cool and seal glassware with foil until you are ready to use it

5.0 Reagents and Standards - General

5.1.1 **NaCl and Na_2SO_4:** Place in shallow tray and heat in muffle oven for 4 hours @ 400 °C (power level 2). Store overnight at 130 °C

5.1.2 **Florisil:** Activate each batch by heating for 16 hours at 130 °C in a glass container loosely covered with foil. Store at 130 °C

5.1.3 **Acidified Na_2SO_4:** Wet down sodium sulfate with enough ether to make it moist (not to saturate it), add 1-2 ml conc. H_2SO_4, mix well and allow to dry. Test pH by adding 1 g acidified Na_2SO_4 to 5 ml DI H_2O, mix well and test with pH paper. pH should be less than 4

5.1.4 **Acid-washed glasswool:** Follow same procedure above for Na_2SO_4. Both of these can be stored in the oven.

5.1.5 **1:1 H_2SO_4:** *Slowly* add one part conc. sulfuric acid to 1 part distilled water. Always add acid to water. This solution is initially very exothermic.

5.1.6 **1:3 H_2SO_4:** *Slowly* add one part conc. sulfuric acid to 3 parts distilled water. This solution is initially very exothermic. Store in the refrigerator.

5.1.7 **10 N NaOH:** Dissolve 400 g solid NaOH pellets to a total volume of 1 liter with reagent-grade water This reaction is very exothermic.

5.1.8 **1:9 HCl:** Slowly add 1 part acid to 9 parts reagent-grade water

6.0 General Quality Control

6.1 Method Blank – General

6.1.1 A **Method Blank** is to be done daily with each batch of samples.

6.1.2 A **Method Blank** must be run with each type of sample i.e. liquid-liquid, w.d., etc.

6.1.2.1 For liq-liq the **Method Blank** consists of 500 ml reagent grade (DI) water with proper amounts of surrogates.
6.1.2.2 For w.d. the method blank consists of the appropriate solvent and surrogates.
6.1.2.3 For SONI, the method blank consists of Na_2SO_4 - 30 g for BNAs and 10 g for pesticides & PCBs.

6.1.3 The method blank should be labeled as such:

MB	eg. **MB**
Type of Ext.	**BNA LIQ-LIQ**
Date Extracted	**10/30/96**

6.2 LCS – Laboratory Control Standards

6.2.1 An LCS and LCS duplicate is analyzed with each extraction batch.

6.2.2 The LCS is reagent water with a known amount of analytes added. It is used to demonstrate that the method, including extraction, is in control.

6.2.3 See specific analytical SOPs for detailed information on LCS concentrations and preparation instructions.

6.3 Surrogates and Matrix Spikes – General

6.3.1 The appropriate surrogate spikes are to be added to each sample including method blanks, duplicates and matrix spikes in the same concentration and volume.
6.3.2 A matrix spike is one form of QA/QC. A matrix spike and duplicate spike are to be done every 20 samples or for each analytical batch and for each type of sample. Matrix spikes are also performed for Appendix IX samples. A matrix spike is added in addition to the normal surrogate spike.
6.3.3 The following are the volumes and concentrations required for organic extraction and matrix spikes: **(The solutions are labeled accordingly and are not interchangeable)**

Acid surrogate	100 µl of 2 mg/ml acid surr. solution
Base/neutral surrogate	100 µl of 1 mg/ml B/N surr. solution
Acid matrix	100 µl of 2 mg/ml acid matrix solution.
Base/neutral matrix	100 µl of 1 mg/ml B/N matrix solution

Pest/PCB surrogate	25 µl of 200 µg/ml Pest/PCB surr. soln. Pest/PCB matrix 25 µl of 200/500 µg/ml Pest/PCB surr. solution
PCB only matrix	50 µl of 100 µg/ml Arochlor 1260 50 µl of 100 µg/ml Arochlor 1016
Herbicide surrogate	100 µl of 100 µg/ml herb surr. solution
Herbicide matrix	10 µl of 100 µg/ml herb matrix solution

7.0 Waste Disposal and Pollution Prevention

7.1 This procedure generates some organic solvent wastes from unused standards and non-aqueous samples and solid waste from solid samples. All waste disposal procedures must comply with federal and local regulations. Refer to the Sample and Waste Disposal SOP A-2050.

8.0 References

8.1 Method 3500, Test Methods for the Analysis of Solid Waste, Physical/Chemical Methods, Update II, Third Edition

8.2 40 CFR Ch1 Pt 136 App. A

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/OE/1550

Volatile Organics Soil Sampling and Screening Method 5035

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 2

SOP Effective Date: 5/31/07

Copy No. _____
Approved By_____
Date _____

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Volatile Organics Soil Sampling and Preparation Using Method 5035

1.0 Scope and Application:

1.1 This SOP describes collection and screening procedures for VOCs in solid materials using SW-846 Method 5035. This SOP is designed for analysis of samples containing low levels of VOC. For high concentrations and oily wastes, the collection procedures of this SOP apply.

1.2 This method uses a hermetically sealed sample vial, the seal of which is never broken from the time of sampling to analysis. Since the sample is sealed, VOC losses during transport are minimized.

1.3 Samples should be screened to prevent contamination of the purge-and-trap system by samples containing high concentrations of purgeable material.

1.4 Since samples are collected in sealed containers, multiple aliquots should be collected to allow for screening and re-analysis.

2.0 Summary of Method:

2.1 VOC samples are collected by taking 5 grams of sample, weighed in the field at time of collection and placing it into a pre-weighed sampling vial. The vial is sealed and shipped to the laboratory, remaining sealed the entire time.

2.2 The vial is placed unopened into the instrument autosampler. Immediately before analysis, reagent water, surrogates, and internal standards are added as applicable without opening the sample vial. The vial is heated to 40°C and the volatiles purged into an appropriate trap.

3.0 Interferences:

3.1 Impurities in the purge gas and from organic compounds out-gassing from plumbing ahead of the trap account for most impurities. Running method blanks will determine if the system is free from contamination. Using PTFE components in the purging device will minimize interferences.

3.2 Samples can be contaminated by diffusion of volatile organics through the septum seal of the sample vial during shipment and storage.

3.3 Carryover contamination can occur when a low concentration sample is analyzed following a high concentration sample. When practical, samples with high concentrations should be followed by analysis of reagent water to check for carryover contamination. If target compounds present in a high concentration sample are found in subsequent samples, it must be demonstrated that the compounds are not due to carryover. If compounds are not found in subsequent samples, then analysis of reagent water is not necessary.

3.4 The laboratory where volatile analysis is performed should be completely free of solvents, especially methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride

4.0 Apparatus and Materials:

4.1 Field Collection: EnCore samplers, or VOC vials with disposable plastic syringes
4.2 Microsyringes – 10-, 25-, 100-, 250-, 500-, and 1000-µL.
4.3 Syringes – 5-mL, gas-tight.
4.4 Balances – Analytical, 0.0001 g, and top-loading, 0.1 g.
4.5 Disposable pipettes – Pasteur.
4.6 Volumetric flasks, Class A – 1-mL, 5-mL and 100-mL, with ground-glass stoppers.
4.7 Spatula – Stainless steel.
4.8 Purge-and-Trap System – Tekmar LSC 3020, or Tekmar LSC 2000
4.9 Pasteur pipets

5.0 Reagents and Standards:

5.1 Organic-free reagent water
5.2 Methanol (CH_3OH) – Purge-and-Trap Grade
5.3 Polyethylene Glycol
5.4 For low concentration samples: Sodium bisulfate: $NaHSO_4$ added to vials prior to shipment to the field.

6.0 Sample Handling and Storage

6.1 Samples collected in the field must be stored on ice at 4°C, packed in appropriate containers, and shipped to the laboratory.

6.2 Once in the laboratory, store samples at 4°C until analysis. Storage area must be free of organic solvent vapors.

6.3 Samples should be analyzed as soon as practical, within the designated holding time from collection (14 days).

7.0 Procedure:

7.1 Preparation of sample vials:

7.1.1 Vials should be prepared in a fixed laboratory or controlled environment, then sealed, and shipped to the field location. Gloves should be worn during preparation.

7.1.2 Add a clean stir bar to each clean vial

7.1.3 Add preservative to each vial prior to shipping to the field. Add approximately 1 g sodium bisulfate to each vial. Adjust based on expected sample size to have approximately 0.2g preservative for each 1g of sample.

7.1.4 Add 5 mL of reagent water to each vial

7.1.5 Seal the vial and add a label to each.

7.1.6 Weigh each prepared vial to the nearest 0.01g and write it on the label.

7.1.7 High concentration soil samples are collected without preservative and are collected and preserved in the field. The following steps outline preparation of vials used for high concentration soil samples.

7.1.7.1 Add 10 mL of methanol to each vial

7.1.7.2 Seal the vial with a screw-cap and septum seal. Add a label to the vial.

7.1.7.3 Weigh the vial to the nearest 0.01 g and write it on the label.

7.1.7.4 Weigh vials containing methanol again in the field before being used. Vials showing loss of methanol should not be used for sample collection.

7.1.8 For oily waste samples, if samples are known to be soluble in methanol or polyethylene glycol, prepare vials the same as high concentration vials. If the solubility is unknown, collect in a standard VOA vials with no preservative.

7.2 For low-concentration soils: collect samples, taking care to minimize the disturbance of the sample to prevent volatile loss. Samples can be transferred using EnCore samplers, purge-and-trap soil samplers, or a cut plastic syringe. Always wear gloves when handling vials.

7.2.1 Collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere, allowing no more than a few minutes at most. Wipe the exterior of the collection device with a clean cloth or towel.

7.2.2 Using the sample collection device, add about 5g of soil to the sample vial. Quickly brush any soil off the vial threads and seal the vial. Store on ice at 4°C.

7.2.2.1 Carbonate minerals in soil may effervesce on contact with acidic preservative. If the effervescence is minimal, loss of VOCs may be minimal if the vial is sealed quickly. Large amounts of effervescence may cause significant loss of VOCs as well as the potential to shatter the sample vial. If carbonates are suspected, collect a test sample to check for effervescence. If significant reaction occurs, collect samples in an unpreserved vial.

7.2.3 Use a portable balance if practical to check that 5g ± 0.5g has been added. The balance should be calibrated in the field.

7.2.4 Alternatively, collect trial samples with a plastic syringe; weigh each sample and note the length of the soil column. Use this to determine the length of soil in the syringe that equals 5g ± 0.5g.

7.2.5 Collect at least two replicate samples in case re-analysis is necessary. Collect in close proximity to the original sample.

7.2.6 An additional aliquot must be collected for screening, dry weight determination, and high concentration analysis (if necessary). This sample should be collected in an unpreserved

VOA vial. If methanol is used for high concentration samples, an additional aliquot is taken in a methanol preserved vial.

7.2.7 If EnCore samplers are used, samples collected in this device should be transferred to soil vials as soon as possible, or analyzed within 48 hours. EnCore samplers should not store samples longer than 48 hours from time of collection.

7.3 For high concentration samples preserved in the field with methanol:

7.3.1 Collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere, allowing no more than a few minutes at most. Wipe the exterior of the collection device with a clean cloth or towel.

7.3.2 Using the sample collection device, add about 5g of soil to the vial containing methanol. Quickly brush any soil off the vial threads and immediately seal the vial. Store on ice at 4°C.

7.3.3 Use a portable balance if practical to check that 5g ± 0.5g has been added. The balance should be calibrated in the field.

7.3.4 Alternatively, collect trial samples with a plastic syringe; weigh each sample and note the length of the soil column. Use this to determine the length of soil in the syringe that equals 5g ± 0.5g.

7.3.5 Other sample weights and volumes of methanol may be used provided that the analyst can demonstrate sensitivity of the overall analytical procedure is appropriate.

7.3.6 Collection of at least one additional sample aliquot is required for dry weight determination. Samples collected in methanol should be clearly labeled as containing methanol, so samples are not analyzed by the closed-system purge-and-trap equipment.

7.4 For high concentration samples not preserved in the field:

7.4.4 Sample collection follows the procedures described above, except that no preservative solution or methanol is in the sample vial. When no preservative is used, larger sample volumes should be collected, filling the vial as full as possible to minimize headspace. A dry weight aliquot is not generally required, but a second sample is useful for screening, and to minimize loss of volatiles in either aliquot.

7.5 For oil waste samples:

7.5.4 If an oily waste is known to be soluble in methanol or polyethylene glycol, the sample can be collected in a vial containing such a solvent as described in section 7.3.

7.5.5 If solubility is unknown, the sample can be collected in an unpreserved vial as outlined in section 7.4. Alternatively, a trial sample can be collected in the field and tested for solubility. If soluble, collect as described in section 7.3.

7.6 Sample Analysis

7.6.4 General purge-and-trap analysis is described in SOP-OA-1000

7.6.5 For low-level soils:

7.6.5.1 Remove the sample vial from storage and warm it to room temperature. Shake the vial gently to ensure that the contents move freely and stirring will be effective. Place the vial in the sample carousel according to manufacturer instructions. Analyze according to SOP-OA-1000.

7.6.6 For unpreserved high-level samples:

7.6.6.1 Mix the contents of the sample by shaking without opening the vial. The sample consists of the entire contents of the sample container. If shaking is not practical, quickly mix the contents with a narrow metal spatula and immediately reseal the vial. Analyze according to SOP-OA-1000

7.6.7 For preserved high-level samples:

7.6.7.1 Weigh the vial to 0.1g as a check on the weight recorded in the field. Add surrogate spikes by injecting through the septum. Shake for 2 minutes to mix. Analyze according to SOP-OA-1000.

7.6.8 For oil waste samples, use SOP-OE-1750 Waste Dilution Extraction.

7.7 If samples require dry weight correction:

7.7.4 Dry weight determination should be done after all samples are analyzed and no additional aliquots will be needed for re-analysis.

7.7.5 Dry weight samples are taken from an unpreserved vial.

7.7.6 Weigh 5-10 g of sample into a pre-weighed beaker or crucible. Record the weight of the beaker and the beaker + sample

7.7.7 Dry this sample overnight at 105°C Allow to cool in a dessicator before weighing.

7.7.8 Weigh the dried sample; subtract the beaker weight from the undried sample + beaker weight and from the dried sample + beaker weight to get the dry sample weight and the original sample weight.

7.7.9 $$\text{Dry weight \%} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

8.0 Quality Control:

All blanks, LCS and matrix spike/spike duplicate should follow the same extraction procedure as for the samples. See SOP-OA-1000 for method specific QC requirements.

7.0 Method Reference:

Method 5035 SW846 December 1996 Revision 0

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/OE/1600

Semi-Volatile Separtory Funnel Liquid/Liquid Extraction

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 3

SOP Effective Date: 5/31/07

Copy No. _____
Approved By _____
Date _____

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1.0 Scope and Application

1.1 This method describes the procedure for separating organic compounds from aqueous samples by extracting with suitable solvent and concentration of the extract for Semi-volatile Organics (SVOCs), also known as Base-Neutral Acids (BNAs).

1.2 Also consult SOP/OE/1500: General Organic Extractions for general extraction information, waste disposal, and health and safety information.

1.3 This extraction procedure is applicable to SW846 (3510C) extractions for SW846 (8270) and EPA 625.

1.4 **NOTE:** EPA Method 625 requires that the acid and base-neutral fractions be kept separate following drying. Once the fractions are extracted, they are analyzed separately. Method 3501C when used for Method 8270 does not require that fractions be separated. When extracting for Method 8270 using 3510C, fractions are combined following drying. See sections 5.6 and 5.7 for specific instructions regarding fractions.

2.0 Summary of Method

2.1 A known volume of sample (if possible, about 1 liter (100 mL for TCLP)) is extracted with Methylene chloride ($MeCl_2$) at pH <2. The sample is then re-extracted with $MeCl_2$ at a pH >11. The extracts are dried, combined and concentrated to 1 mL for semi-volatile analysis.

3.0 Apparatus and Materials

3.1 Separatory funnels - 0.5 L, 1.0 L and 2.0 L with polytetrafluorethylene (PTFE) stop-cock.

3.2 Kuderna-Danish (KD) apparatus:

- 3.2.1 Concentrator tubes - 10 mL graduated
- 3.2.2 Evaporation Flask - 500 mL
- 3.2.3 Snyder Column - three ball macro
- 3.2.4 Snyder Column - two ball micro
- 3.2.5 Clips

3.3 Solvent vapor recovery system

3.4 Boiling chips

3.5 Water bath

3.6 Crimp top vials - 2 mL

3.7 pH indicator paper

3.8 Syringes - 1 mL and 0.5 mL; For EPA 625 use 500 uL syringe

3.9 Graduated cylinder - 1 L

3.10 Erlenmeyer Flask - 250 mL

4.0 Reagents

4.1 All reagents used are reagent-grade chemicals as certified by the manufacturer.

4.2 Organic free water or fluid extracts for TCLP

4.3 Sodium hydroxide solution (10 N) 40 g of NaOH dissolve in 100 mL of H_2O

4.4 Sulfuric acid solution, 1:1 solution of H_2SO_4 in water

4.5 Anhydrous Sodium sulfate (Na_2SO_4)- purify by heating at 65 °C overnight; rinsed with methylene chloride prior to use.

4.6 Reagent-grade methylene chloride ($MeCl_2$)

5.0 Procedure

5.1 Measure 1 liter (100 mL for TCLP) of sample using a graduated cylinder. Different volumes of sample may be used for various types of analysis.

5.2 Add 20 µl of semi-volatile surrogate standards (See Table I for concentration) into all blanks, matrix spike/matrix spike duplicates (MS/MSD), LCS and samples.

5.3 Select one **sample** (not fluid extracts or DI water) for MS/MSD for each analytical batch of 20 samples or less. Add 20 µl each of acid and base-neutral matrix spike standards (See Table I for concentration) into each of the MS and MSD samples.

5.4 With each analytical batch, extract a method blank(organic-free water), or a fluid extract for TCLP,with appropriate amount of surrogate added as per Section 5.2.

5.5 Also with each analytical batch, extract a Laboratory Control Sample (LCS) (organic-free water) or fluid extract with surrogate as per Section 5.2 and matrix spike as per Section 5.3.

5.5.1 For Method 625 the Acid and Base-Neutral standards and spikes must be combined prior to beginning the first extraction step. The fractions will be separated during the extraction procedure in the same manner as normal samples.

5.6 Perform two separate $MeCl_2$ extractions on the sample first at basic pH and then at acid pH.

5.6.1 For semi-volatiles, first adjust the pH of the sample to >11, with 10 N sodium hydroxide, and extract sample as per Section 5.6.2. Then adjust pH to <2, with 1:1 sulfuric acid, and extract sample as per Section 5.6.2. Then Proceed to Section 5.7.

5.6.2 Add 60 mL of $MeCl_2$. Seal and shake the separatory funnel vigorously for 1-5 minutes with periodic venting. Allow the organic layer to separate for 10 minutes and collect the bottom $MeCl_2$ layer in an Erlenmeyer flask. If the sample produces an emulsion, use mechanical techniques to disrupt the emulsion, such as stirring and filtration through glass wool. Repeat the extraction two more times, using 60 mL of $MeCl_2$ each time. Combine all three organic layers. NOTE: Keep the acid and base-neutral $MeCl_2$ extracts separate until they are combined following drying, as described in Section 5.7.4. **If Method 625 is being used acid and base-neutral fractions should be kept separate. For Method 625 proceed to section 5.8. For Method 3510C, proceed to Section 5.7.**

5.7 Dry, Combine and Concentrate Sample Extracts (Method SW846(3510C):

5.7.1 Assemble a KD concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask.

5.7.2 Attach the solvent vapor recovery glassware to the Snyder column.

5.7.3 Dry the acid extract by passing through 10-cm deep drying column containing anhydrous sodium sulfate. Rinse the acid extract Erlenmeyer flask with 10-20 mL of $MeCl_2$ and transfer through the drying column into the concentrator tube.

5.7.4 Repeat the steps in Section 5.7.3 using the base-neutral extract.

5.7.5 Add a boiling chip to the evaporation flask and attach a three ball Snyder column. Place the KD apparatus in a hot water bath. Adjust the position of the apparatus and temperature of the bath to complete the concentration in 10-20 min. When the volume is reduced to approximately 3 mL, remove the KD apparatus and allow it to cool for 5-10 min. Remove the

Snyder column and rinse the flask and its lower joints with $MeCl_2$ into the concentrator tube.

5.7.6 Add another clean boiling chip to the concentrator tube and attach a two ball micro Snyder column. Place the concentrator tube in the water bath to a depth that will complete the concentration in about 5-10 minutes. When the apparent volume reaches approximately 0.5 mL, remove the KD apparatus from the water bath and allow it to cool. Adjust the final extract volume to 1 mL with $MeCl_2$ and transfer the extract to a 2 mL autosampler vial.

5.7.7 Cap and label the vial as follows:

FIRSTECHNOLGY #
BNA
L/L
TCLP or ORIG
DATE

5.8 Dry and Concentrate Separate Fraction Extracts (Method EPA 625):

5.8.1 Assemble a KD concentrator for the acid fraction by attaching a 10-mL concentrator tube to a 500-mL evaporation flask. Assemble another KD concentrator for the basic fraction. Get an Erlenmeyer flask for each fraction.

5.8.2 Attach the solvent vapor recovery glassware to the Snyder columns.

5.8.3 Dry the acid extract by passing through 10-cm deep drying column containing anhydrous sodium sulfate. Rinse the acid extract Erlenmeyer flask with 10-20 mL of $MeCl_2$ and transfer through the drying column into the acid fraction concentrator tube.

5.8.4 Repeat the steps in Section 5.8.3 using the base-neutral extract.

5.8.5 Add a boiling chip to each evaporation flask and attach a three ball Snyder column to each. Place each KD apparatus in a hot water bath. Adjust the position of the both and the temperature of the bath to complete the concentration in 10-20 min. When the volume is reduced to approximately 3 mL, remove each KD apparatus and allow them to cool for 5-10 min. Remove the Snyder columns and rinse each flask and its lower joints with $MeCl_2$ into its designated concentrator tube.

5.8.6 Add another clean boiling chip to each concentrator tube and attach a two ball micro Snyder column to each tube. Place the concentrator tubes in the

water bath to a depth that will complete the concentration in about 5-10 minutes. When the apparent volume reaches approximately 0.5 mL, remove each KD apparatus from the water bath and allow them to cool. Adjust the final fraction extract volume to 1 mL with $MeCl_2$ and transfer each fraction to a separate 2 mL autosampler vial.

5.8.7 Cap and label the vial as follows:

FIRSTECHNOLGY #
BN (or ACID)
L/L
TCLP or ORIG
DATE

6.0 Quality Control

6.1 All blanks, matrix spike, matrix spike duplicate, LCS and QC extracted samples should follow the same analytical procedure as followed for samples.

6.2 Additional QC performed by laboratory:

6.2.1 At least once per month a special QC standard is extracted and carried through the entire analytical sequence. This QC standard contains all the compounds usually included in the analytical instrument calibration. It can be obtained from either the calibration standard or the calibration check standard stocks.

7.0 Method References

7.1 SW846 (3510C) December 1996 Revision 3

7.2 EPA Method 625 (40CFR Part 136)

TABLE I

	Standard Concentration (µg/mL)
Semi-volatile Acid Surrogates Standard:	10000
Semi-volatile Base-Neutral Surrogates Standard:	5000
Semi-volatile Acid Matrix Spike Standard/LCS:	10000
Semi-volatile Base-Neutral Matrix Spike Standard/LCS:	5000
Semi-volatile QC Standard:	200

These concentrations are typical of standards purchased by FIRSTECHNOLOGY. Concentrations may vary if other manufacturer's standards are purchased.

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/OE/1700

Semi-Volatile Extraction by Sonication

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 2

SOP Effective Date: 5/31/07

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Approved By	_____
Date	_____

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1.0 Scope and Application

1.1 This method is used for extraction of semi-volatile organic compounds from soils, sludges and waste material.

1.2 Also consult SOP/OE/1500: General Organic Extractions for general extraction information, waste disposal, and health and safety information.

2.0 Summary of Method

2.1 A known amount of sample (usually 10-30 g.) is mixed with anhydrous sodium sulfate to form a free flowing sample. This is extracted with Methylene chloride ($MeCl_2$) + Acetone. The extract is dried and concentrated to 1 mL for semi-volatile analysis.

3.0 Apparatus and Materials

3.1 Ultrasonic disrupter – must be a horn type device of a minimum of 300 watts power with pulsing capability; Use a ¾" horn size for this method.

3.2 Beakers - 400 mL

3.3 Kuderna-Danish (KD) apparatus.

- 3.3.1 Concentrator tubes - 10 mL graduated
- 3.3.2 Evaporation Flask - 500 mL
- 3.3.3 Snyder Column - three ball macro
- 3.3.4 Snyder Column - two ball micro
- 3.3.5 Clips.

3.4 Solvent vapor recovery system
3.5 Boiling chips
3.6 Water bath
3.7 Crimp top vials - 2 mL
3.8 Syringes – 20 uL
3.9 Laboratory pan balance
3.10 400 mL beaker
3.11 Whatman No. 41 filter paper
3.12 Conical glass funnel

4.0 Reagents:

4.1 All reagents used are reagent-grade chemicals as certified by the manufacturer
4.2 Organic-free water
4.3 Anhydrous Sodium Sulfate (Na_2SO_4)- purify by heating at 400 °C for 4 hours
4.4 Reagent-grade $MeCl_2$ (methylene chloride)
4.5 Reagent-grade Acetone
4.6 50:50 Mix of Acetone and Methylene Chloride. Add an equal part acetone to an equal part of methylene chloride; make to an appropriate volume.

5.0 Procedure:

5.1 Weigh 10-30 g of well mixed sample into a 400-mL beaker.
5.2 Add 30-60 g anhydrous sodium sulfate. Mix well to form a free-flowing mixture.
5.3 Add 20 µL semi-volatile surrogate standards (See Table I for concentration) into all blanks, matrix spike/matrix spike duplicates (MS/MSD), LCS and samples.
5.4 Select one sample for MS/MSD for each analytical batch of 20 samples or less. Add 20 µL each of acid and base-neutral matrix spike standards (See Table I for concentration) into each of the MS and MSD samples.
5.5 With each analytical batch, extract a method blank (organic-free, clean sand) with an appropriate amount of surrogate added as per Section 5.3.
5.6 Also with each analytical batch, extract a Laboratory Control Sample (LCS) (organic-free, clean sand) with an appropriate amount of surrogate added as per Section 5.3 and matrix spikes as per Section 5.4.
5.7 Immediately add 100 mL of 50:50 $MeCl_2$/Acetone. Place bottom surface of the tip of sonicator horn about 1/2" below the surface of solvent but above the solid sample layer. Sonicate for 3 min. Decant the extract and filter through a cone of Whatman No. 41 filter paper into a 500-mL flask.
5.8 Repeat extraction/sonication two more times (100 mL $MeCl_2$ each time), decanting solvent after each extraction. After the final extraction, pour the entire sample into the Whatman filter cone and rinse with extraction solvent.
5.9 Dry and Concentrate Sample Extracts

5.9.1 Assemble a KD concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask.
5.9.2 Attach the solvent vapor recovery glassware to the Snyder column.
5.9.3 Dry the extract by passing through 10-cm deep drying column containing anhydrous sodium sulfate. Rinse the 400 mL beaker with 10-20 mL of 50:50 $MeCl_2$/Acetone and transfer through the drying column into the concentrator tube.
5.9.4 Add a boiling chip to the evaporation flask and attach a three ball Snyder column. Place the KD apparatus in a hot water bath. Adjust the position of the apparatus and temperature of the bath to complete the concentration in 10-20 min. When the volume is reduced to approximately 3 mL,

remove the KD apparatus and allow it to cool for 5-10 min. Remove the Snyder column and rinse the flask and its lower joints with $MeCl_2$ into the concentrator tube.

5.9.5 Add another clean boiling chip to the concentrator tube and attach a two ball micro Snyder column. Place the concentrator tube in the water bath to a depth that will complete the concentration in about 5-10 minutes. When the apparent volume reaches approximately 0.5 mL, remove the KD apparatus from the water bath and allow it to cool. Adjust the final extract volume to 1 mL with $MeCl_2$ and transfer the extract to a 2 mL autosampler vial.

5.9.6 Cap and label the vial as follows:

FIRSTECH. #
BNA
S/L
TCLP or ORIG
DATE

5.10 If samples require dry weight correction:

5.10.1 Weigh 5-10 g of sample into a pre-weighed beaker or crucible. Record the weight of the beaker and the beaker + sample.

5.10.2 Dry this sample overnight at 105°C. Allow to cool in a dessicator before weighing.

5.10.3 Weigh the dried sample; subtract the beaker weight from the undried sample + beaker weight and from the dried sample + beaker weight to get the dry sample weight and the original sample weight.

5.10.4 $$\text{Dry weight \%} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

6.0 Quality Control

6.1 All blanks, matrix spike, matrix spike duplicate, LCS and QC extracted samples should follow the same analytical procedure as followed for samples.

7.0 Method References

7.1 SW846(3550B) December 1996 Revision 3.

TABLE I

	Standard Concentration (µg/mL)
Semi-volatile Acid Surrogates Standard:	2000
Semi-volatile Base-Neutral Surrogates Standard:	1000
Semi-volatile Acid Matrix Spike Standard/LCS:	2000
Semi-volatile Base-Neutral Matrix Spike Standard/LCS:	1000
Semi-volatile QC Standard:	200

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/OE/1900

Pesticide/PCB Separatory Funnel
Liquid/Liquid Extraction

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 2

SOP Effective Date: 5/31/07

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1.0 Scope and Application

1.1 This method describes the procedure for separating organic compounds from aqueous samples by extracting with suitable solvent and concentration of the extract for PCBs and/or Pesticides.

2.0 Summary of Method

2.1 A known volume of sample (if possible, about 1 liter) is extracted with Methylene chloride ($MeCl_2$) at a pH between 5 and 9. The extract solvent is exchanged for hexane and concentrated to 1 mL for PCB and/or pesticide analysis.

3.0 Apparatus and Materials

3.1 Separatory funnels - 0.5 L, 1.0 L and 2.0 L with polytetrafluorethylene stop-cock.

3.2 Kuderna-Danish (KD) apparatus:

3.2.1 Concentrator tubes - 10 mL graduated

3.2.2 Evaporation Flask - 500 mL

3.2.3 Snyder Column - three ball macro

3.2.4 Snyder Column - two ball micro

3.2.5 Springs

3.3 Solvent vapor recovery system.

3.4 Boiling chips

3.5 Water bath

3.6 Crimp top vials - 2 mL

3.7 pH indicator paper

3.8 Syringes - 1 mL and 0 5 mL

3.9 Graduated cylinder - 1 L

3.10 Erlenmeyer Flask - 250 mL

4.0 Reagents

4.1 All reagents used are reagent-grade chemicals as certified by the manufacturer

4.2 Organic free water

4.3 Sodium hydroxide solution (10 N): 40 g of NaOH dissolved in 100 mL of H_2O

4.4 Sulfuric acid solution, 1:1 solution of H_2SO in water.

4.5 Anhydrous Sodium sulfate (Na_2SO_4)- purify by heating at 400 °C for 4 hours

4.6 Reagent-grade $MeCl_2$ and hexane

5.0 Procedure

5.1 Measure 1 liter of sample using a graduated cylinder. Different volumes of sample may be used for various types of analysis.

5.2 Add 25 µl of PCB/Pesticides surrogate standards (See Table I for concentration) into all blanks, matrix spike/spike duplicates (MS/MSD), LCS and samples.

5.3 Select one sample for MS/MSD for each analytical batch of 20 samples or less. Add 50 µl for PCBs and 25 µl for pesticides matrix spike standards (See Table I for concentration) into each of the MS and MSD samples.

5.4 With each analytical batch, extract a method blank (organic free water) spiked with appropriate amount of surrogate as per Section 5.2.

5.5 Also with each analytical batch, extract a Laboratory Control Sample spiked with surrogate as per Section 5.2 and matrix spike as per Section 5.3.

5.6 For PCB/Pesticides analysis, adjust pH to between 5 and 9, if needed, by using 1:1 sulfuric acid or 10 N sodium hydroxide.

5.7 Add 60 mL of $MeCl_2$. Seal and shake the separatory funnel vigorously for 1-5 min with periodic venting. Allow the organic layer to separate for 10 min and collect the bottom $MeCl_2$ layer in an Erlenmeyer flask. If the sample produces an emulsion, use mechanical techniques to disrupt the emulsion, such as stirring and filtration through glass wool. Repeat the extraction two more times, using 60 mL of $MeCl_2$ each time. Combine all three organic layers.

5.8 Dry, Exchange Solvent and Concentrate Sample Extract:

5.8.1 Assemble a KD concentrator by attaching 10mL concentrator tube to a 500 mL evaporation flask.

5.8.2 Attach the solvent vapor recovery glassware to the Snyder column.

5.8.3 Dry the extract by passing through 10 cm deep drying column containing anhydrous sodium sulfate. Rinse the Erlenmeyer flask with 10-20 mL of $MeCl_2$ and transfer into the concentrator tube.

5.8.4 Add a boiling chip to the evaporation flask and attach a three ball Snyder column. Place the KD apparatus in a hot water bath. Adjust the position of the apparatus and temperature of the bath to complete the concentration in 10-20 min. When the volume is reduced to approximately 3 mL, remove the KD apparatus and allow it to cool for 5-10 min.

5.8.5 Add 40 mL of hexane for solvent exchange and concentrate to about 10 mL. Remove the Snyder column and rinse the flask and its lower joints with hexane into the concentrator tube.

5.8.6 Add another clean boiling chip to the concentrator tube and attach a two ball micro Snyder column. Place the concentrator tube in the water bath to a depth that will complete the concentration in about 5-10 minutes. When the apparent volume reaches approximately 3 mL, remove the KD apparatus from the water bath and allow it to cool.

5.8.7 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube. Adjust the final volume to 5.0 mL.

5.8.8 Transfer 1 mL of the final extract to a 2 mL autosampler vial for PCBs/Pesticide analysis.

5.8.9 Cap and label as follows:

FIRSTECH. #
PCB or PEST or PEST/PCB
L/L
TCLP or ORIG
DATE

6.0 Quality Control

6.1 All blanks, matrix spike, matrix spike duplicate, LCS and QC extracted samples should follow the same analytical procedure as followed for samples. See analytical SOPs SOP/OA/1300 and SOP/OA/1500 for additional details.

7.0 Method Reference

7.1 SW846(3510C) December 1996 Revision 3.

7.2 EPA Method 608 (40CRF Part 136)

TABLE I

	Standard Concentration (µg/mL)
PCBs/Pesticides Surrogate Standard:	100
PCB Matrix Spike Standard/LCS (1260 Aroclor):	100
PCB Matrix Spike Standard/LCS (1016 Aroclor)	100
Pesticide Matrix Spike Standard/LCS:	200/500

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/OE/1950

Cleanup Procedures for Pesticide and PCB Extracts

Approved By: _____, Lab Manager
Mark Kalmeyer

Approved By: _____, QA/QC Officer
Monique Posner

Revision: 0

SOP Effective Date: 10/11/06

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1.0 Scope and Application

1.1 This method describes procedures for performing cleanup of samples in hexane extracts for analysis of Polychlorinated Biphenyls (PCBs) and/or Pesticides.

1.2 Three procedures are described here: acid cleanup, copper cleanup for sulfur contamination, and florisil cleanup.

2.0 Summary of Method

2.1 For acid cleanup, a sample extract in hexane is mixed with aqueous sulfuric acid. The layers are separated via centrifuge, effecting removal of impurities into the acid phase. **Acid cleanup is not used if pesticide analysis is required.**

2.2 For copper cleanup: elemental sulfur has a solubility similar to many organochlorine pesticides; as such, sulfur contamination follows along with pesticides through normal extraction and cleanup. Copper powder is used to remove sulfur interference.

2.3 For florisil cleanup, a pesticide sample extract in hexane is passed through a column of florisil. The eluant is concentrated as necessary for pesticide analysis. **Florisil cleanup is not used for PCB analysis.**

3.0 Apparatus and Materials

3.1 Acid Cleanup/Copper Cleanup

3.1.1 Bench-top Centrifuge

3.1.2 Glass transfer pipets

3.1.3 15 mL screw-top glass culture tubes (Teflon-lined caps)

3.2 Florisil Cleanup

3.2.1 Kuderna-Danish (KD) apparatus:

3.2.1.1 Concentrator tubes - 10 mL graduated
3.2.1.2 Evaporation Flask - 500 mL
3.2.1.3 Snyder Column - three ball macro
3.2.1.4 Snyder Column - two ball micro
3.2.1.5 Springs

3.2.2 Solvent vapor recovery system.

3.2.3 Boiling chips

3.2.4 Water bath

3.2.5 Crimp top vials - 2 mL

3.2.6 15 mL screw-top glass culture tubes (Teflon-lined caps)

4.0 Reagents

4.1 Reagent grade chemicals shall be used in all tests. Unless otherwise specified, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water, ASTM Type 1 reagent water.

4.3 Acid Cleanup

4.3.1 Concentrated Sulfuric Acid

4.4 Copper Cleanup

4.4.1 Nitric Acid, HNO_3, dilute.

4.4.2 Solvents, pesticide grade or equivalent

4.4.2.1 Acetone, CH_3COCH_3
4.4.2.2 Hexane, C_6H_{14}
4.4.2.3 2-Propanol, $CH_3CH(OH)CH_3$

4.4.3 Copper powder – Remove oxides by treating with dilute nitric acid, rinse with organic-free reagent water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.

4.4.4 Tetrabutylammonium (TBA) sulfite reagent

4.4.4.1 Tetrabutylammonium hydrogen sulfate, $[CH_3(CH_2)_3]_4NHSO_4$.

4.4.4.2 Sodium sulfite, Na_2SO_3.

4.4.4.3 Prepare reagent

4.4.4.3.1 Dissolving 3.39 g of tetrabutylammonium hydrogen sulfate into 100 mL of organic-free reagent water.

4.4.4.3.2 Extract this solution three times with 20 mL portions of hexane.

4.4.4.3.3 Discard the hexane extracts, and add 25 g sodium sulfite to the resulting solution.

4.4.4.3.4 Store the saturated sodium sulfite solution in an amber bottle with a PFTE-lined screw cap. This solution can be stored at room temperature for at least one month.

4.5 Florisil Cleanup

4.5.1 6% ether in hexane (ratio of 6 mL ether to 94 mL hexane)

4.5.2 15% ether in hexane (ratio of 15 mL ether to 85 mL hexane)

4.5.3 50% ether in hexane (ratio of 50 mL ether to 50 mL hexane)

4.5.4 Florisil

4.5.5 Anhydrous Sodium Sulfate (Na_2SO_4) – purify by heating at 400°C for 4 hours.

5.0 <u>Procedure</u>

5.1 Acid Cleanup for PCB Extracts

5.1.1 Transfer PCB sample extract (in hexane) to a 15-mL glass culture tube (if not already in such a tube).

5.1.2 Add approximately 2 mL concentrated sulfuric acid. Mix via shaking by hand for 1-2 minutes.

5.1.3 Centrifuge until aqueous and organic layers are separated.

5.1.4 Draw off the top hexane layer.

5.1.5 Transfer 1 mL of extract to autosampler vial.

5.1.6 Cap and label the vial as described in the original sample preparation procedure.

5.2 Florisil Cleanup of Pesticide Sample Extracts

5.2.1 Plug a 10-mL disposable pipet with glass wool. Pack the column with florisil to a volume of 5 cc (above the glass wool). Add about 0.5 cc Na_2SO_4 on top of the florisil.

5.2.2 Place an evaporator flask and KD under the florisil column. Rinse the column with enough hexane to completely wet the florisil and to allow some of the hexane to drip into the KD flask.

5.2.3 Pipet sample (in hexane) into the florisil column, letting it pass through to the KD flask. Rinse the sample container (usually a culture tube) with 2-3 mL hexane and pipet into the column following the original sample.

5.2.4 Pipet the following mixtures into the florisil column, in sequence, collecting eluants in the KD evaporator flask:

5.2.4.1 30 mL 6% ether in hexane

5.2.4.2 30 mL 15% ether in hexane

5.2.4.3 30 mL 50% ether in hexane

5.2.5 Drain and discard the column.

5.2.6 Attach a 3-ball Snyder column to the evaporator flask and add a boiling stone.

5.2.7 Concentrate the sample to a final volume of 5 mL.

5.2.8 Transfer 1 mL of extract to an autosampler vial.

5.2.9 Cap and label the vial as described in the original sample preparation procedure.

5.3 Copper Cleanup Procedures

5.3.1 Following sample concentration, if only PCB analysis is required, copper cleanup may be preceded by an acid cleanup. If pesticide is required proceed straight to copper cleanup.

5.3.2 If sulfur crystallization occurs, centrifuge to settle the crystals, and carefully draw off the sample extract with a disposable pipette, leaving the excess sulfur in the concentration vessel. Transfer 1 mL of the extract to a calibrated centrifuge tube.

5.3.3 Add a small amount of copper powder to the centrifuge tube. Centrifuge to separate the phases.

5.3.4 Separate the extract from the copper by drawing off the extract with a disposable pipette and transfer 1 mL to an autosampler vial.

5.3.5 Cap and label the vial as described in the original sample preparation procedure.

6.0 Quality Control

6.1 All blanks, matrix spike, matrix spike duplicate, LCS and QC extracted samples should follow the same analytical procedure as followed for samples.

7.0 Method Reference

1.1 EPA Method 608 (40CRF Part 136)

1.2 SW846(3600B) December 1996 Revision 3.

1.3 SW846(3660B) December 1996 Revision 2.

1.4 SW846(3620B) December 1996 Revision 2.

1.5 SW846(3665A) December 1996 Revision 1.

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/OE/2000

Pesticide/PCB Extraction by Sonication

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 2

SOP Effective Date: 6/4/07

Copy No. _____
Approved By_____
Date _____

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1.0 Scope and Application:

This method is used for extraction of semi-volatile and PCBs/Pesticides organic compounds from soils, sludges and waste material.

2.0 Summary of Method:

Weigh 10-30 grams of sample and mix with anhydrous sodium sulfate to form a free flowing sample. This is extracted three times with the appropriate solvent and concentrated using KD technique.

3.0 Apparatus and Materials:

3.1. Sonicator
3.2. KD apparatus (see method)

4.0 Reagents:

4.1. All solvents used are reagent grade.
4.2. Organic free reagent-grade water.
4.3. Anhydrous Sodium Sulfate (Na_2SO_4). Purify by heating at 400 °C for 4 hours.
4.4. Methylene Chloride ($MeCl_2$)
4.5. Acetone
4.6. Hexane

5.0 Procedure:

5.1. Weigh 10-30 g (to the nearest 0.1 g) of well mixed sample into a 600-mL beaker.
5.2. Add 30-60 g of anhydrous sodium sulfate.
5.3. Prepare QC samples: For Method Blank and Laboratory Control Sample (LCS), use 10-30 g of clean sand. For MS/MSD, select one sample for each analytical batch of 20 samples or less. Use 5.0 g of sample; add either 50 µL PCB matrix spike standard (1260/1016) or 25 µL Pesticide matrix spike standard into each of the MS and MSD samples. For LCS, prepare both a PCB LCS (with 50 µL PCB matrix spike standard (1260/1016) and a Pesticide LCS (with 25 µL Pesticide matrix spike standard). PCB spikes and LCS standards must consist of PCB-1016 and 1260.
5.4. Add 25 µl of PCB/Petsicide surrogate into all samples, MS/MSDs, Lab Control Sample (LCS) and blank.
5.5. Immediately add 300 mL of 1:1 methylene chloride/acetone solvent mixture.
5.6. Place bottom surface of the tip of horn about 1/2" below the source of solvent but above the sample layer.
5.7. Sonicate for 6 minutes.

5.8. Dry the extract by passing through 10-cm deep funnel containing anhydrous sodium sulfate into a concentrator tube. Rinse the beaker with 10-20 mL of $MeCl_2$ and transfer through funnel into the concentrator tube. Rinse funnel with 10-20 mL of $MeCl_2$.
5.9. Repeat sonication extraction at least two more times.
5.10. KD concentrations, follow SOP for PCB/Pesticide Liquid/Liquid Extractions.
5.11. Transfer 1 mL of extract to 2-mL autosampler vial.
5.12. Cap and label as follows:

FIRSTECH #
Pest/Soni, PCB/Soni or P/P Soni
Date Ext.

5.13. If samples require dry weight correction:

5.14. Weigh 5-10 g of sample into a pre-weighed beaker or crucible. Record the weight of the beaker and the beaker + sample.

5.15. Dry this sample overnight at 105°C. Allow to cool in a dessicator before weighing.

5.16. Weigh the dried sample; subtract the beaker weight from the undried sample + beaker weight and from the dried sample + beaker weight to get the dry sample weight and the original sample weight.

5.17. Dry weight % = $\frac{\text{g of dry sample}}{\text{g of sample}} \times 100$

5.18.

6.0 Quality Control:

All blanks, matrix spike, matrix spike duplicate, LCS and QC extracted samples should follow the same analytical procedure as followed for samples.

7.0 Method Reference:

7.1 SW846(3550B) December 1996 Revision 2.

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/M/1000

Metals Sample Preparation

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 2

SOP Effective Date: 5/31/07

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Copy No. _____
Approved By_____
Date _____

1.0 Scope and Application

1.1 This document details sample preparation procedures for various metals analysis techniques

1.2 Covers preparation techniques for ICP-ES, ICP-MS, graphite furnace atomic absorption, and flame atomic absorption.

1.3 Matrices covered include water, wastewater, soils, sediments, sludge, oils, paints, organic material, wipe samples, air filter samples, and paint chips.

2.0 Summary

2.1 See specific digestion procedures.

3.0 Definitions

3.1 ICP-ES: Inductively Coupled Plasma Emission Spectroscopy

3.2 ICP-MS: Inductively Coupled Plasma Mass Spectroscopy

3.3 GFAA: Graphite Furnace Atomic Absorption

3.4 FLAA: Flame Atomic Absorption

4.0 Environmental Health and Safety

4.1 Laboratory coats, gloves, and safety glasses shall be worn in order to avoid exposure to strong acids and other reagents used for most of these procedures

4.2 Due to potential toxicity of reagents and the strong acids used for most acid digestions, all digestion procedures must be preformed in a fume hood.

4.3 Care should be taken during digestions when heating is involved using hot plates, block digesters and other heat sources.

5.0 Equipment and Supplies

5.1 See specific digestion procedures.

6.0 Reagents and Standards

6.1 See specific digestion procedures

7.0 Quality Control

7.1 See specific analytical SOPs to determine required quality control for sample preparation.

8.0 Index of Methods

8.1 *Method A*: Sample Preparation and Acid Digestion of Aqueous Samples for Total Metals Analysis by ICP-ES, ICP-MS, FLAA, or GFAA.

8.2 *Method B*: Sample Preparation and Filtration of Aqueous Samples for Dissolved Metals Analysis by ICP-ES, ICP-MS, FLAA, or GFAA.

8.3 *Method C*: Sample Preparation and Acid Digestion of Soils, Sediments, and Sludges for Total Metals Analysis by ICP-ES, ICP-MS, FLAA, or GFAA.

8.4 *Method D*: Sample Preparation and Acid Digestion of Oils, Paints, and Organic Materials for Total Metals Analysis by ICP-ES, ICP-MS, FLAA, or GFAA.

8.5 *Method E*: Sample Preparation and Acid Digestion of Wipe Samples for Total Metals Analysis by ICP-ES, ICP-MS, FLAA, or GFAA

8.6 *Method F*: Sample Preparation and Acid Digestion of Air Filters for Total Metals Analysis by ICP-ES, ICP-MS, FLAA, or GFAA.

8.7 *Method G*: Sample Preparation and Acid Digestion of Paint Chips for Total Metals Analysis by ICP-ES, ICP-MS, FLAA, or GFAA

9.0 Procedure

The following procedures describe preparation procedures for metals analysis:

SAMPLE PREPARATION AND ACID DIGESTION OF AQUEOUS SAMPLES FOR TOTAL METALS ANALYSIS BY ICP-ES, ICP-MS, FLAA, and GFAA

METHOD A (SW846 3010A-M)

A-1 REAGENTS

1. Water, reagent-grade
2. HNO3, concentrated (trace pure or better)
3. HCl, concentrated (trace pure or better)

A-2 EQUIPMENT

1. 50 ml digestion tubes or 250 ml Griffin beakers (Note: If Boron is one of the requested elements, this digestion procedure must be done in a Teflon beaker)
2. Hotplate or heating block set to 95 °C.
3. Whatman filter paper (#41) and filter funnel
4. Graduated cylinder.
5. Ribbed watch glass.

A-3 PROCEDURE FOR HEATING BLOCK DIGESTION

1. This procedure can be used for non-organic samples in this classification.
2. Transfer a 50 mL aliquot of well mixed sample into a digestion tube.
3. Add 2.5 ml concentrated HNO_3 and 2.5 ml concentrated HCl and place in the heating block
4. Heat samples in heating block until the volume has been reduced to 25 ml. DO NOT boil sample
5. Cool sample to room temperature. If the sample contains insoluble material that might clog the nebulizer, filter with Whatman #41 filter paper
6. Bring the sample up to a final volume of 50 ml with reagent-grade water

A-4 PROCEDURE FOR HOTPLATE DIGESTION

1. Transfer a 50 ml aliquot of well mixed sample to a beaker.
2. Add 2.5 ml concentrated HNO_3 and 2.5 ml concentrated HCl. Place the beaker on the hotplate and cover with a ribbed watch glass
3. Heat sample until the volume has been reduced to 15-20 ml DO NOT boil sample.
4. Cool sample to room temperature. If the sample contains insoluble material that might clog the nebulizer, filter with Whatman #41 filter paper.
5. Bring the sample to a final volume of 50 ml with reagent-grade water.

A-5 PROCEDURE FOR SAMPLE DIGESTION WHEN ANALYSIS TO BE PERFORMED BY ICP-MS

1. This section should be used when analyzing for all metals except antimony. When antimony is to be analyzed, one of the above digestions (with HCl and HNO_3) should be used.
2. Follow all the steps listed in either the hotplate/beaker or heating block procedure above, with the following modification: do not add HCl; add only 2.5 ml of HNO_3 prior to the heating/volume reduction step.

A-5 PROCEDURE FOR SAMPLE DIGESTION WHEN ANALYSIS TO BE PERFORMED BY GFAA

1. Not using HCl in digestion increases the life of graphite components during analysis. This section should be followed when only GFAA will be used for analysis, and antimony is not required. When antimony is to be analyzed by GFAA, or other analytical methods will be used on the digested samples, one of the above digestions (with HCl and HNO_3) should be used

2. Follow all the steps listed in either the hotplate/beaker or heating block procedure above, with the following modification: do not add HCl; add only 2.5 ml of HNO_3 prior to the heating/volume reduction step.

SAMPLE PREPARATION AND FILTRATION OF AQUEOUS SAMPLES FOR DISSOLVED METALS ANALYSIS BY ICP-ES, ICP-MS, FLAA, and GFAA

METHOD B (SW-846 3005A-M)

B-1 REAGENTS

1. Water, reagent-grade
2. HNO_3, concentrated

B-2 EQUIPMENT

1. 250 ml Griffin beakers
2. Whatman filter paper (#45)
3. Millipore pressurized filter apparatus
4. Plastic sample holding bottles.

B-3 PROCEDURE

1. Sample should be filtered through a #45 filter at the time of collection; the liquid phase should then be acidified with nitric acid.
2. If the sample is received unfiltered, filter through a #45 filter using the Millipore pressurized filter device located in the TCLP room and then acidify the liquid phase with nitric acid.
3. If total and dissolved metals are required, shake the sample and separate into two aliquots. Acidify the first aliquot with nitric acid and digest as per the method. Filter the second aliquot and then acidify the filtrate.
4. Record the dissolved samples in the digestion book as being a filtered sample.
5. The filtering apparatus should be cleaned with soap, water, and a DI water rinse between each sample. In addition, a new filter needs to be replaced after each sample.
6. Follow with appropriate digestion procedure prior to analysis.

SAMPLE PREPARATION AND ACID DIGESTION OF SOILS, SEDIMENTS AND SLUDGES FOR TOTAL METALS ANALYSIS BY ICPES, ICP-MS, FLAA OR GFAA

METHOD C (SW846 3050B-M)

C-1 REAGENTS

1. Water, (nanopure)
2. HNO_3, concentrated (Tracepure grade or better)
3. HCl, concentrated (Tracepure grade or better)
4. H_2O_2, 30% solution

C-2 EQUIPMENT

1. 50 ml digestion tubes or 250 ml Griffin beakers (Note: If Boron is one of the requested elements, this digestion procedure must be done in a Teflon beaker.)
2. Hotplate or heating block set to 95 °C
3. Whatman filter paper (#41) and filter funnel
4. Graduated cylinder
5. Ribbed watch glass

C-3 PROCEDURE FOR HEATING BLOCK DIGESTION

1. Weigh between 1.00 to 4.00 g of sample into a 50 ml digestion tube. Measure to nearest 0.01 g.
2. Add 5 ml H_2O, 2.5 ml HNO_3 and 2.5 ml of HCl to sample. Slurry the mixture.
3. Place the tube in the heating block and cautiously evaporate to 10 ml. DO NOT boil the sample or allow it to become dry.
4. Remove digestion tube(s) from heating block and let them cool. Slowly add 2 mL of H_2O_2 and return to the heat until effervescence has stopped.
5. Remove digestion tube from the heating block and cool to room temperature.
6. Filter out any solid particles which may clog the sample introduction system. Bring the final volume to 50 ml with reagent grade water.

C-4 PROCEDURE FOR HOTPLATE DIGESTION

1. Weigh between 1.00 to 4.00 g of sample into a beaker. Measure to nearest 0.01 g.
2. Add 10 ml H_2O, 5 ml HNO_3 and 5 ml of HCl to sample. Slurry the mixture.
3. Place the beaker on the hotplate and cover with a ribbed watch glass.
4. Heat sample until the volume has been reduced to 15-20 ml. DO NOT boil sample. Cool sample to room temperature.
5. Filter with Whatman #41 filter paper.
6. Bring the sample to a final volume of 50 ml with reagent-grade water.

C-5 PROCEDURE FOR SAMPLE DIGESTION WHEN ANALYSIS TO BE PERFORMED BY ICP-MS

1. This section should be used when analyzing for all metals except antimony. When antimony is to be analyzed, one of the above digestions (with HCl and HNO_3) should be used.
2. Follow all the steps listed in either the hotplate/beaker or heating block procedure above, with the following modification: do not add HCl; add only 2.5 ml of HNO_3 prior to the heating/volume reduction step.

C-6 PROCEDURE FOR SAMPLE DIGESTION WHEN ANALYSIS TO BE PERFORMED BY GFAA

3. Not using HCl in digestion increases the life of graphite components during analysis. This section should be followed when only GFAA will be used for analysis, and antimony is not required. When antimony is to be analyzed by GFAA, or other analytical methods will be used on the digested samples, one of the above digestions (with HCl and HNO_3) should be used.
4. Follow all the steps listed in either the hotplate/beaker or heating block procedure above, with the following modification: do not add HCl; add only 2.5 ml of HNO_3 prior to the heating/volume reduction step.

C-7 DRY WEIGHT DETERMINATION

1. Weigh 5-10 g of sample into a pre-weighed beaker or crucible. Record the weight of the beaker and the beaker + sample.

2. Dry this sample overnight at 105°C. Allow to cool in a dessicator before weighing.

3. Weigh the dried sample; subtract the beaker weight from the undried sample + beaker weight and from the dried sample + beaker weight to get the dry sample weight and the original sample weight.

4. Dry weight % = $\frac{\text{g of dry sample}}{\text{g of sample}} \times 100$

SAMPLE PREPARATION AND ACID DIGESTION OF OILS, PAINTS, AND ORGANIC MATERIAL FOR TOTAL METALS ANALYSIS BY ICPES, ICP-MS, FLAA OR GFAA

METHOD D (SW846 3050M)

D-1 REAGENTS

1. Water, (nanopure)
2. HNO_3, concentrated (Tracepure grade or better)
3. HCl, concentrated (Tracepure grade or better)
4. H_2O_2, 30% solution

D-2 EQUIPMENT

1. 50 ml digestion tubes or 250 ml Griffin beakers (Note: If Boron is one of the requested elements, this digestion procedure must be done in a Teflon beaker.)
2. Hotplate or heating block set to 95 °C
3. Whatman filter paper (#41) and filter funnel
4. Graduated cylinder
5. Ribbed watch glass

D-3 PROCEDURE FOR HEATING BLOCK DIGESTION

1. Transfer a 5.0 ml aliquot of well mixed sample into a digestion tube.
2. Add 5 ml H_2O, 2.5 ml HNO_3 and 2.5 ml of HCl to sample. Slurry the mixture.
3. Place the tube in the heating block and cautiously evaporate until no fumes are given off. DO NOT boil the sample or allow it to become dry.
4. Remove digestion tube(s) from heat and allow them to cool.
5. Slowly add 2 ml H_2O_2 and heat until effervescence has stopped. Return to heat.
6. Remove digestion tube from the heating block and cool to room temperature.
7. Filter out any solid particles that may clog the sample introduction system. Bring the final volume to 50 ml with reagent grade water.

D-4 PROCEDURE FOR HOTPLATE DIGESTION

1. Transfer a 5.0 ml aliquot of well mixed sample into a beaker.
2. Add 5 ml H_2O, 2.5 ml HNO_3 and 2.5 ml of HCl to sample. Slurry the mixture.
3. Place the beaker on the hotplate and cover with a ribbed watch glass.
4. Heat sample until the volume has been reduced to 15-20 ml. DO NOT boil sample.
5. Slowly add 2 ml H_2O_2 and heat until effervescence has stopped.
6. Cool sample to room temperature. Filter with Whatman #41 filter paper.
7. Bring the sample to a final volume of 50 ml with reagent-grade water.

SAMPLE PREPARATION AND ACID DIGESTION OF WIPE SAMPLES FOR LEAD ANALYSIS BY ICP-ES, ICP-MS, FLAA OR GFAA METHOD E (SW846 3050B-M)

E-1 REAGENTS

1. Water, (nanopure)
2. HNO_3, concentrated (Tracepure grade or better)

E-2 EQUIPMENT

1. 50 ml digestion tubes
2. Heating block set to 95 °C
3. Whatman filter paper (#41) and filter funnel
4. Graduated cylinder

E-3 PROCEDURE FOR HEATING BLOCK DIGESTION

1. Place entire wipe (baby wipe or filter) into a digestion tube.
2. Add 10 ml H_2O, and 2.5 ml HNO_3. Slurry the mixture.
3. Place the tube in the heating block.
4. Digest sample for approximately 15 min. DO NOT boil the sample or allow it to become dry.
5. Remove digestion tube from the heating block and cool to room temperature.
6. Bring the final volume to 50 ml with reagent grade water (leaving wipe/filter in the digestion tube.) If the wipe/filter disintegrates to generate material that might clog the instrument injection system, the sample should be filtered through Whatman #41 filter paper

SAMPLE PREPARATION AND ACID DIGESTION OF AIR FILTERS FOR TOTAL METALS ANALYSIS BY ICPES, ICP-MS, FLAA OR GFAA METHOD F (SW846 3050B-M)

This method can be used to for NIOSH 7300 and NIOSH 7082 for lead analysis. NIOSH 7300 and 7082 should be reported with an M to indicate a modification to the printed methods.

F-1 REAGENTS

1. HNO_3, concentrated (trace pure or better)
2. Water, reagent grade

F-2 EQUIPMENT

1. 50 ml digestion tube
2. Heating block set to 95 °C

F-3 PROCEDURE

1. Open the cassette filter holder and transfer the filter into an empty digestion tube.
2. Add 2.5 ml of HNO_3 to the digestion tube. Filter should start dissolving.
3. Place the tube in the heating block and heat until the filter is dissolved.
4. Remove the tube from the heating block and allow it to cool.
5. Dilute to 25 ml with reagent-grade water.

F-4 QUALITY CONTROL

1. A method blank should be run with each batch of samples. To prepare a method blank, add a new, unused filter to a digestion tube and proceed as instructed in step F-3.
2. An LCS/LCS dup should be prepared with each sample batch. To prepare, add a new, unused filter to a digestion tube. Pipet a volume of liquid lead solution onto the filter so that contact is made with the filter media. Proceed as instructed in step F-3.

SAMPLE PREPARATION AND ACID DIGESTION OF PAINT CHIPS FOR TOTAL METALS ANALYSIS BY ICP-ES, ICP-MS, FLAA, or GFAA

METHOD G (SW846 3050B-M)

G-1 REAGENTS

1. Water, (nanopure)
2. HNO_3, concentrated (Tracepure grade or better)
3. HCl, concentrated (Tracepure grade or better)
4. H_2O_2, 30% solution

G-2 EQUIPMENT

1. 50 ml digestion tubes or 250 ml Griffin beakers (Note: If Boron is one of the requested elements, this digestion procedure must be done in a Teflon beaker.)
2. Hotplate or heating block set to 95 °C
3. Whatman filter paper (#41) and filter funnel
4. Graduated cylinder
5. Ribbed watch glass
6. Analytical balance

G-3 PROCEDURE FOR HEATING BLOCK DIGESTION

1. Weigh 0.100 to 0.500 g of sample into a digestion tube.
2. Add 5 ml H_2O, 2.5 ml HNO_3
3. Place the tube in the heating block and heat for approximately 20 minutes until orange fumes disappear. DO NOT boil the sample or allow it to become dry.
4. Remove digestion tube from the heating block and cool to room temperature.
5. Slowly add 3ml H_2O_2. Return tube to heating block and heat for 5 minutes after effervescence has stopped.
6. Remove digestion tube from the heating block and cool to room temperature.
7. Add 2.5ml HCl. Return tube to heating block and heat for an additional 15 to 20 minutes until brown fumes disappear.
8. Remove digestion tube from the heating block and cool to room temperature.
9. Filter out any solid particles that may clog the sample introduction system. Bring the final volume to 50 ml with reagent grade water.

G-4 PROCEDURE FOR HOTPLATE DIGESTION

1. Weigh 0.100 to 0.500 g of sample into a beaker
2. Add 5 ml H_2O, 2.5 ml HNO_3.
3. Place the beaker on the hotplate and cover with a ribbed watch glass.
4. Reflux for approximately 20 minutes until orange fumes disappear DO NOT boil the sample or allow it to become dry.
5. Remove beaker from the hotplate and cool
6. Slowly add 3ml H_2O_2. Return beaker to hotplate and heat for 5 minutes after effervescence has stopped.
7. Remove beaker from the hotplate and cool.
8. Add 2.5ml HCl. Return beaker to hotplate and heat for an additional 15 to 20 minutes until brown fumes disappear.
9. Remove beaker from the hotplate and cool.
10. Rinse the cover glass with reagent-grade water in a way such that the washings are combined with the digested sample
11. Filter out any solid particles which may clog the sample introduction system Bring the final volume to 50 ml with reagent grade water

10.0 Waste Management and Pollution Prevention

10.1 This procedure generates acidic wastes. All wastes shall be disposed of according to local regulatory as well as laboratory-established guidelines. Refer to the Laboratory's Disposal SOP A-2050.

10.2 For pollution prevention, the laboratory should digest samples with volumes consistent with laboratory use to minimize the amount of acidic waste generated.

11.0 References

11.1 Test Methods for Evaluating Solid Wastes, SW-846, Third Edition, Update III, July 1992, Method 7470A.

11.2 Sample Waste Disposal SOP, A-2050

11.3 Ohio EPA Laboratory Manual for Chemical Analyses of Public Drinking Water, 2000

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/M/1500

Metals Analysis by Inductively Coupled Plasma Emission Spectrometry (ICP-ES)

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 2

SOP Effective Date: 5/31/07

Controlled Copy on Green Paper

Copy No. _____
Approved By_____
Date _____

Metals Analysis by Inductively Coupled Plasma Emission Spectrometry (ICP-ES)

1.0 Methods

1.1 This SOP meets the requirements of the following methods: 200 7, SW6010, SM3125 and NIOSH 7300

2.0 Applicable Matrices

2.1 This method applies to a wide range of environmental matrices including drinking, surface, and saline waters, mobility extracts, soils, sludges, sediments, oils and domestic and industrial waste water.

2.2 This SOP applies to lead analysis in airborne particulates, dust wipes, paint chips, and soils See Appendix B for specific requirements to meet AIHA guidelines.

2.3 For drinking water analysis, see Appendix A for specific requirements to meet Ohio EPA drinking water guidelines.

3.0 Detection Limits

3.1 Reporting limits vary per element, and detection limit studies are performed on an annual basis See QA/QC documentation for currently calculated detection limits

4.0 Scope and Application

4.1 Inductively Coupled Plasma Emission Spectrometry (ICP-ES) is used to determine metals and some non-metals in solution

4.2 This SOP is applicable to the following elements:

Table 4.1

Analyte	Symbol	Analyte	Symbol
Aluminum	Al	Mercury	Hg
Antimony	Sb	Molybdenum	Mo
Arsenic	As	Nickel	Ni
Barium	Ba	Phosphorous	P
Beryllium	Be	Potassium	K
Boron	B	Selenium	Se
Cadmium	Cd	Silica	SiO_2
Calcium	Ca	Silver	Ag
Chromium	Cr	Sodium	Na
Cobalt	Co	Strontium	Sr
Copper	Cu	Thallium	Tl
Iron	Fe	Tin	Sn
Lead	Pb	Titanium	Ti
Lithium	Li	Vanadium	V
Magnesium	Mg	Zinc	Zn
Manganese	Mn		

The sensitivity and optimum linear ranges for each element will vary with wavelength, matrix, and operating conditions. See Appendix C for recommended wavelengths Additional elements may be analyzed if a method validation is performed prior to analysis.

4.3 ICP-ES may be used for analysis of dissolved analytes after suitable filtration and acid preservation. Dissolved solids should be <0.2% (w/v).

4.4 With the exception of silver, where this method may be used for drinking water, samples may be analyzed directly without digestion if the turbidity is <1 NTU at the time of analysis

4.5 The method cited is dependent on the sample matrix. Methods are assigned to samples during login. See section 1.0 for a list of methods covered by this SOP.

4.6 For drinking water analysis, see Appendix A for additions/modifications to the normal procedures outlined in this SOP.

4.7 This SOP can be used to test for lead in paint, soil, dust, and air. For requirements to fulfill AIHA certification guidelines, see Appendix B.

4.8 MDL studies are performed annually. See the latest QA/QC documentation that details the most recent MDL studies and reporting limits

5.0 Summary

5.1 An aliquot of a well mixed, homogenous aqueous or solid sample is accurately weighed or measured for sample processing. See specific procedures for sample processing

5.2 An ICP source consists of a flowing stream of argon gas ionized by an applied radio frequency typically oscillating at 27.1 MHz. The field is inductively coupled to the ionized gas by a water cooled coil surrounding a quartz torch that supports and confines the plasma.

5.3 A sample aerosol is generated in an appropriate nebulizer and spray chamber and is carried into the plasma at 6000-8000 K. Ionization of a high percentage of atoms produces an ionic emission spectra. This efficient, high-temperature dissociation significantly reduces chemical interferences and results in low detection levels for many elements

5.4 The light emitted from the ICP is focused onto the entrance slit of monochromator. A precisely aligned exit slit isolates a portion of the spectrum for an intensity measurement with a photomultiplier tube. These measurements are processed by computer software to produce analyte concentrations

6.0 Definitions

6.1 Calibration Blank: reagent-grade water and instrumental grade acids combined in the same concentrations as in the standards.

6.2 Calibration Standard (CAL STD): solution containing known concentrations of the analytes to be analyzed.

6.3 Continuing Calibration Blank (CCB): same as ICB except it is run *during* or *after* a batch of samples

6.4 Continuing Calibration Verification (CCV): same as ICV except it is run *during* or *after* a batch of samples.

6.5 Dissolved Analyte: the concentration of an aqueous sample that will pass through a 0.45 um membrane filter prior to sample acidification.

6.6 Duplicate sample (DUP): an intralaboratory split sample, for which both aliquots are brought through the entire digestion and analysis procedure

6.7 Initial Calibration Blank (ICB): the calibration blank is DI water analyzed as a sample following the calibration

6.8 Initial Calibration Verification (ICV): a primary source standard solution (same as the **CAL STD**) containing known concentrations of the analytes to be analyzed. Run immediately following initial calibration

6.9 ICSA - Interference Check Standard A: a solution, containing potential interfering elements Al, Ca, Fe, Mg, Na, K, in phosphoric/sulfuric/hydrochloric/citric acids utilized in the determination of interferences and the need for correction with ICP-AES analyses

6.10 ICSAB - Interference Check Standard A and B: a solution containing all the components of ICSA, spiked with metals (As, Cd, Cr, Co, Cu, Mn, Ni, Ag, Zn) to be checked for interference effects to be used in the determination of intereferences and the need for correction with ICP-AES analyses.

6.11 Lab Control Sample (LCS): a matrix-based material of known analyte concentration, or reagent water spiked with analyte standard, brought through the entire digestion and analysis process. If such material is not available, reagent-grade water or the closest available matrix-based material, spiked with an appropriate amount of analyte should be used

6.12 Lab Control Sample (LCS2): a second source matrix-based standard analogous to LCS.

6.13 Linear Dynamic Range (LDR): the concentration range through which the analytical curve remains linear.

6.14 Lower Linearity Check Sample: a standard solution with a concentration at or close to that of the lower end of the linear calibration range

6.15 Matrix Spike (MS) and Spike Duplicate (MSD): a known aliquot of sample that is spiked with a known amount of the CAL STD concentration. These are then brought through the entire sample preparation and analysis process.

6.16 Method Blank (MB): analyte-free matrix-based material brought through the entire sample preparation as a sample and analyzed as though it were a sample. If such material is not available, reagent-grade water or the closest available matrix-based material should be used.

6.17 Reference Standard (RS): a secondary source standard solution containing a known concentration of the analytes to be analyzed in a different concentration than the CAL STD and prepared from a different source. Analyzed after initial calibration and ICV to verify calibration

6.18 Secondary Source: a source of standards used that is independent of the source used for calibration. This should be from a different vendor unless it can be determined that the vendor used different standard lots to prepare the standards. Different assigned vendor lot numbers are not sufficient to determine true independence. Consult traceability documentation or contact the manufacturer.

6.19 Reporting Limit Check (RLC): (also called Detection Limit Check) a standard prepared at a concentration equal to the reporting limit

6.20 Interference Check Standard (ICS): used to determine possible interferences during analysis.

6.21 Total Recoverable Analytes: concentration of an analyte determined by direct analysis (analysis of a solution extract of a solid or an unfiltered aqueous sample following digestion with hot, dilute mineral acids)

7.0 Typing Conventions

7.1 **Keynames**. All computer keys or buttons, including function keys and monitor keys, appear in small capitals and should be pressed when bracketed as <SUCH>

7.2 **Italics**. *ITALICS* indicate words and characters you type and take effect by pressing <ENTER>.

7.3 **Monitor character string selection**. All character strings to be selected from the monitor, either with a mouse click on an icon or menu selection, or with a designated soft-key or direction arrows and <ENTER> appear in small capitals and are DOUBLE UNDERLINED.

8.0 Interferences

8.1 Interferences can be 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.

8.2 Background emission can be compensated for by subtracting the background emission determined by measurement(s) adjacent to the wavelength peak, or by using an alternate wavelength.

8.3 Spectral overlaps can be avoided by using alternate wavelengths, using interelement correction, or measuring the interfering elements.

8.4 When not using interelement correction, interference check standards must be analyzed to verify the absence of interelement spectral interference

8.5 Certain physical characteristics can cause interferences in the sample nebulization and transport processes. Changes in viscosity or suface tension, high dissolved solids, or high acid concentrations can cause physical interferences. Effects can be reduced by dilution, high-solids nebulizer, a peristaltic pump, or an internal standard element.

8.6 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. These are normally not significant, but can be eliminated by buffering the sample, matrix matching, or by standard additions.

8.7 Memory interferences can by caused by carryover from one sample to the next. It is avoided by having sufficient rinse time with a rinse blank between samples

9.0 Environmental Health and Safety

9.1 Due to potential toxicity of reagents, all digestion procedures must be preformed in a fume hood.

9.2 Laboratory coats, gloves, and safety glasses shall be worn in order to avoid exposure to strong acids and reducing agents used in this procedure.

9.3 The plasma should only be viewed with proper protection from ultraviolet emissions.

9.4 When opening the torch compartment for maintenance, be sure all parts are cool before attempting to remove or touch parts.

10.0 Equipment and Supplies

10.1 Inductively Coupled Plasma Emission Spectrometer: FIRSTECH, Inc. uses a Varian Liberty Sequential ICP-EX, which includes the following:

10.1.1 Computer controlled spectrometer: Varian Liberty Series Plasma 96 software

10.1.2 Quartz axial plasma torch.

10.1.3 Nebulizer and spraychamber.

10.1.4 Peristaltic Pump: 2 channel, variable speed, computer controlled

10.1.4.1 Peristaltic pump tubing:

10.1.5 Radio Frequency Generator (FCC compliant).

10.1.6 SPS-5 Autosampler.

10.2 High purity liquid argon

10.3 Pipettes, Adjustable, Microliter with disposable tips Sizes can range from 5 to 5,000 µL, as required Pipette tips used should be suitable for trace metal analysis.

10.4 Volumetric Flasks, Class A, assorted sizes including 100 and 1000 mL.

10.5 Volumetric pipettes, Class A, assorted sizes from 0.5 to 5 mL.

10.6 Digestion tubes, 50 mL

10.7 Graduated cylinders, Class A, various sizes from 10 to 1000 mL.

10.8 Acid dispensing bottles, polyethylene, polypropylene or equivalent material, in various dispensing sizes (1, 2, 5, and 10 mL recommended).

11.0 Reagents and Standards

11.1 Nitric acid (concentrated): Acid must be analyzed to determine levels of impurities. Used in sample prep, rinse water, blanks, and preservation of standards

11.2 Hydrochloric acid (concentrated): Acid must be analyzed to determine levels of impurities Used in sample prep, rinse water, blanks, and preservation of standards.

11.3 30% Hydrogen Peroxide (H_2O_2): Used in digestion of solids and oil samples

11.4 Concentrated Calibration Standard Cal1 (Ca, Mg, K, Na: 5000 ug/mL, Al, Ba: 2000 ug/mL, Co, Mn, Ni, V, Zn: 500 ug/mL, Cu, Ag: 250 ug/mL, Be: 50 ug/mL): obtained from Inorganic Ventures or an equivalent source.

11.5 Concentrated Calibration Standard Cal2 (Sb: 1000 ug/mL): obtained from Inorganic Ventures or an equivalent source

11.6 Concentrated Calibration Standard Cal3 (As, Pb, Se, Tl: 1000 ug/mL, Cd: 500 ug/mL): obtained from Inorganic Ventures or an equivalent source.

11.7 LCS: prepared during sample prep; add 0.25 mL each of Cal1 and Cal3 + any additional required elements

11.8 MS/MSD: prepared during sample prep; add 0.25 mL each of Cal1 and Cal3 + any additional required elements.

11.9 Concentrated Single Element Standards (1000ppm): Mo, Sb, Sn, Sr; obtained from various manufacturers.

11.10 Reference Standards, ICS100-1(Be: 50 ug/mL, Cd, Zn: 150 ug/mL, Pb: 500 ug/mL, Mn: 100 ug/mL, Se: 200 ug/mL), ICS100-2 (Ba, Co, Cu, V: 100 ug/mL, Fe: 10,000 ug/mL), ICS100-3 (As: 500 ug/mL, Mo, Si: 100 ug/mL), ICS100-4 (Al, Na: 200 ug/mL, Ca: 1000 ug/mL, Cr, Ni: 20 ug/mL, K: 400 ug/mL), ICS100-5 (Ag: 50 ug/mL, B: 100 ug/mL, Mg: 1000 ug/mL, Sb, Tl: 200 ug/mL): used for preparation of RS standard, obtained from Environmental Express or a similar source.

11.11 Concentrated interference check standard CLPP-ICS-A: obtained from Inorganic Ventures or an equivalent source.

11.12 Calibration Standards and QC Samples:

11.12.1 Primary source standards shall be used to prepare the initial calibration curve standards and continuing calibration verification (CCV) samples. A calibration blank is also used for calibration.

11.12.2 Calibration Standard: 5 mL each of Cal1 and Cal3 (Inorganic Ventures); 5 mL each of Mo, Sb (or Cal2), Sn; 1 mL Sr. Bring up to 1 L volume in a solution of 5% nitric and 5% hydrochloric acid.

11.12.3 ICV/CCV: 5 mL each of Cal1 and Cal3 (Inorganic Ventures); 5 mL each of Mo, Sb (or Cal2), Sn; 1 mL Sr. Bring up to 1L volume in a solution of 5% nitric and 5% hydrochloric acid.

11.12.4 RS Standard: 2 mL of ICS100-1, 1 mL of ICS100-2, 2 mL of ICS100-3, 2 mL of ICS100-4, 2 mL of ICS100-5, 2 mL of Sn, 1 mL of Sr. Bring up to 200 mL volume in a solution of 5% nitric and 5% hydrochloric acid.

11.12.5 ICSA: 5 mL of CLPP-ICS-A, bring up to 100 mL in a solution of 5% nitric and 5% hydrochloric acid.

11.12.6 ICSAB: 5 mL of CLPP-ICS-A, 0.5 mL each of Cal1 & Cal2, 1 mL each of Mo, Sb, Sn, 0.25 mL of Sr. Bring up to 100 mL in a solution of 5% nitric and 5% hydrochloric acid.

12.0 Sample Containers, Preservation, and Handling

12.1 All sample containers must be pre-washed with detergents, acids, and reagent water. Plastic and glass containers are both suitable.

12.2 Aqueous samples must be acidified to a pH of <2 with nitric acid. Samples requiring dissolved metals should be filtered and prior to acidification. Holding times for metals samples is six months.

12.3 Non-aqueous samples shall be refrigerated and analyzed within six months.

13.0 Quality Control

13.1 The analyst must demonstrate proficiency in performing the analysis. Method proficiency must be re-demonstrated anytime a major method modification is made, a major software revision is added, or a major instrument modification is made. Demonstration of method proficiency may also be required after major instrument maintenance. This is decided on a case-by-case basis through discussions with the Laboratory Technical Manager and Quality Assurance Manager.

13.2 An initial demonstration of instrument performance should be performed before analyzing samples. Performance of an MDL study should be done prior to using this method. MDL studies should be done annually, or when major changes are made to equipment or procedures.

13.3 A preparation batch of samples is defined as a group of up to twenty field samples of similar matrix type that have been prepared at the same time or time sequence with the same lots of reagents for the same analysis. In addition to the twenty samples, each preparatory batch will contain at a minimum, a method blank, a laboratory control sample, a matrix spike, a matrix spike duplicate and a matrix duplicate. Preparation batches of samples may be continuously strung together in these run sequences, as long as the analytical batch QC requirements meet the acceptance criteria established within the appropriate SOP.

13.4 An analytical or instrumental batch is defined as samples that are analyzed together within the same time period or in continuous sequential time periods. Within the analytical batch are included individual QC requirements as defined by the analytical (determinative) method.

13.5 As per manufacturers specifications, calibration is performed using a single point calibration. The calibration is followed by ICV, RS, an ICB, and an RLC (drinking water only). If any of these check standards fail, the instrument must be recalibrated. See Section 18 for acceptance criteria.

13.6 Run an ICSA and ICSB to determine possible interferences from certain elements.

13.7 At least every six months; analyze a high-range standard to determine the linear dynamic range of analytes. A range can be chosen based on typical values seen during analysis. Values should be within 10% of true values; lower concentrations until measured values are within 10%.

13.8 For specific elements (As, Se, Tl, Zn) that are prone to common interference, multiple wavelengths are always run to help determine if an interference is present; if results for both wavelengths do not agree closely (±15%), further investigation is needed to determine if there is a reportable result. First, examination of spectral data should be employed. Dilution and post-digestion spikes may be used as described in section 13.9. As a final resort, ICP-MS may be used to confirm positive hit results by ICP.

13.9 For new or unusual matrices, or when interference checks and multiple wavelength analysis may indicate a matrix interference problem, the following procedures can be helpful:

13.9.1 Serial Dilution: a 1:5 dilution of the sample should agree to ±10% for actual positive sample hits. If not, there is a chance an interference is present.

13.9.2 Post-digestion spike: spike a digested sample as per section 11.8. If necessary, sample may be diluted prior to spiking. Recovery should be 75-125% of the known value or a matrix interference may be present.

14.0 Calibration and Standardization

14.1 If the torch has been removed, a torch alignment must be performed. The alignment is done with a 1 ppm Manganese solution prior to analysis.

14.2 A calibration is prepared using a single point calibration curve, as per Varian recommendations.

14.3 Following calibration, several check standards are evaluated to establish the integrity of the calibration. These include, in the following order: ICV, RS (±10% of true value), ICB and Lower Linearity Check Sample. See section 18 for quality control acceptance criteria.

15.0 Procedure

15.1 Instrument Set-Up

15.1.1 Turn on vent

15.1.2 Make sure gas pressure is adjusted to 60 psi

15.1.3 Check to see that the main power on the ICP and the computer are on

15.2 Start-Up

15.2.1 Fit the tubing in the peristaltic pump.

15.2.2 If the Liberty software is closed, double click on the LIBERTY icon

15.2.3 Open the INSTRUMENT page. Turn on the pump. Turn on the Plasma (using IGNITION page or by pressing <Shift><F4>) Allow instrument to warm up approximately 30 minutes

15.2.4 If the torch has been changed or moved, a torch alignment must be performed. Go to the TORCH ALIGN page Introduce a 1 ppm Mn solution and then do the scan: Click on HORIZONTAL, then on INTENSITY. When the scan is done, repeat for VERTICAL and again for HORIZONTAL (a second time). The instrument will automatically adjust the torch to the optimal position.

15.2.5 Go to the W/L CALIBRATION page and click on CALIBRATE to perform an initial wavelength calibration. Once it is complete, check to see that the "Diff to Last" column is ± 10 for all wavelengths (0.00 may be any value). If any of the values exceeds ± 10, repeat the calibration. Close the INSTRUMENT page.

15.3 Sample Analysis

15.3.1 Click on WORKSHEET

15.3.2 Select NEW FROM. Choose the current template file; the name of the current template filename is taped to the instrument computer monitor; current file is "icptemplate3"

15.3.3 Enter the name of the data file (ex.: Metals0309) then click OK.

15.3.4 Go to the "Develop" page of the worksheet. Click EDIT METHOD.

15.3.5 Using the ELEMENTS CONDITIONS STANDARDS and QCP pages, make any corrections to the method. Then, under FILE, choose UPDATE, then EXIT, then YES. The method is loaded.

15.3.6 On the LABELS page, enter sample/QC labels for each sample to be analyzed with dilution factors. Choose injection mode: MANUAL or SPS AUTOSAMPLER. (If autosampler is used, adjust the sequence parameters Rack Type No. 1 to the proper size, 25 x 60). Change the total sample number, if necessary.

15.3.7 On the WORKSHEET Page, highlight all elements that need to be run for each sample.

15.3.8 To start a manual run, click the GREEN ARROW and introduce samples as indicated.

15.3.9 To start an automatic run, make sure the standards and samples are loaded properly in the autosampler rack. Check the tubing to make sure it is connected. Be sure the SPS Autosampler is turned on and has rinse water. Click the GREEN ARROW.

15.3.10 Before running samples, QC parameters must be checked. In order, following the calibration sample, run the following samples, checking that all QC criteria are met before any samples are analyzed: ICV, RS (±10% of true value), ICB and Lower Linearity Check Sample (drinking water only).

15.3.11 Interference checks with solutions SIC/ICSA/ICSAB-I, -II and -III must be run once per week (following the initial 5-day series to verify interference correction factors) for all elements, except AIHA lead samples. For AIHA lead samples the SIC/ICS-I, -II and -III must be performed daily. If soil, paint, wipes or air filter samples are analyzed for lead, the SIC/ICS-I, -II and -III for lead must also be reanalyzed at the end of the batch.

15.3.12 A matrix-specific MB and LCS must be run once per batch and a minimum frequency of 5%. For each batch and at a minimum frequency of 5%, there must be a duplicate and a spike/spike duplicate. A CCB and a CCV (±10% of true value) must be run after each batch and at a minimum frequency of 10%.

15.3.13 For all QC parameters listed above, if control limits are not achieved, the problem must be identified and necessary QC samples repeated until control limits are met. All samples affected by non-compliant QC must be reanalyzed. See section for acceptance limits and corrective actions.

15.3.14 At the end of the run, print the report (Under FILE - PRINT).

15.4 Shut-Down

15.4.1 Once the run is complete, turn off the plasma (click the PLASMA OFF icon or press <F4> or go to the IGNITION page and select PLASMA OFF)

15.4.2 Release tubing

15.4.3 Turn off vent.

15.4.4 Go to INSTRUMENT SETUP page

15.4.5 Go to DIAGNOSTICS

15.4.6 Click on GASBOX, make sure MONO PURGE is checked.

15.4.7 Turn off computer screen only.

16.0 Calculations

16.1 The concentration of an element (µg/L) in the sample is read from the calibration curve or directly from the instrument if operating in the direct concentration mode. Samples that required dilution are adjusted according to the following equation:

$$\text{element } (\mu g/L) = A \times \left(\frac{B}{C}\right)$$

where:

A = sample concentration from calibration curve
B = volume (mL) of calibration blank used to dilute sample
C = volume (mL) of sample used

16.2 The concentration of metals in solid matrices is reported in mg/kg of waste as follows:

mg/kg of an element = A(V/W)

where:

A = final concentration read from calibration curve in µg/L
V = Final volume of processed sample (L) (0.1 L recommended)
W = Dry weight of sample digested (g)

16.3 Calculation of Recovery for MS/MSD and LCS analyses.

$$\%\text{Recovery} = \frac{C_s - C_u}{C_n} \times 100$$

where:

C_s = Measured concentration of the spiked sample aliquot
C_u = Measured concentration of the unspiked sample aliquot (use 0 for LCS analyses)
C_n = Nominal (or theoretical) concentration of the spiked sample aliquot

16.4 Calculation of Precision

$$\%\text{RPD} = \frac{(C_1 - C_2)}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where:

C_1 = Measured concentration of the first sample
C_2 = Measured concentration of the second sample

16 5 Correlation coefficient

$$R = \frac{\sum XY}{\sqrt{\sum X^2 \sum Y^2}}$$

where:
R = correlation coefficient
X = standard concentration
Y = absorbance

17.0 Method Performance

17.1 See QA/QC documentation, which lists the latest MDL study results and method performance data.

17.2 Consult control charts where available to track method performance.

17.3 Also see original test methods, Method 200.7 EMMC Version Rev 4.4, Section 13.0, SW-846 Method 6010 Section 13 0,

18.0 Data Assessment and Corrective Actions

Table 18.1 QC Acceptance Criteria

Standard	Acceptance Criteria			
	SW846 6010	EPA 200.7	CLP	SM3120
Instrument Blank (initial)	Zero	Zero	Zero	Zero
ICS-AB	80-120%	80-120%	80-120%	N/A
RLC	70-130%	70-130%	70-130%	70-130%
ICV	80-120%	90-110%	80-120%	90-110%
ICB	Below reporting limit	Below reporting limit	Below reporting limit	Below reporting limit
RS	90-110%	95-105%	90-110%	95-105%
MB	Below reporting limit	Below reporting limit	Below reporting limit	Below reporting limit
LCS	80-120%	85-115%	80-120%	85-115%
MS/MSD (Recovery)	80-120% (50-150% TCLP)	70-130%	75-125%	80-120%
MS/MSD (RPD)	<20%	N/A	<20%	N/A
Duplicates	RPD = <20%	RPD = <20%	RPD = <20%	RPD = <25% if <20x MDL, RPD = <10% if >20x MDL
CCV	90-110%	90-110%	90-110%	95-105%
CCB	Below reporting limit	Below reporting limit	Below reporting limit	Below reporting limit

18 1 If the initial calibration verification (including detection limit check, ICV, ICB, and RS) fails, repeat and/or make fresh standards as needed. Do not run any samples until acceptable results are obtained

18.2 If the continuing calibration verification (including CCB and CCV) fails:

18 2 1 Repeat and/or make fresh standards as needed.

18.2.2 Adjust calibration or redo calibration if the above doesn't work

18 2 3 If calibration was adjusted or redone, when QC is acceptable, re-analyze all samples since the last acceptable continuing calibration verification.

18.3 If the Method Blank or LCS fails, repeat the analysis once.

18.4 If the MS/MSD fails:

18.4.1 Repeat the analysis once; if results are still unacceptable, proceed to Step 18.4.2.

18.4.2 If sample greater than 4x the amount of spiking standard added, no further post-digestion spiking is necessary. If sample less than 4x amount of spike, proceed to 18.4.3.

18.4.3 Perform a post digestion spike and spike duplicate. If results are still unacceptable, proceed to 18.4.4

18.4.4 Dilute sample, preferably to give results above detection limit. Repeat 18 4 3. Continue repeating steps 2 and 3 until acceptable results are obtained.

19.0 Contingencies for Out-of-Control and Unacceptable Data

19.1 Most analytical data has been checked for acceptability during analysis. Analysts should flag any unacceptable data, make any relevant notes as to the cause, or record any observations noted that may assist in the evaluation of the data.

19.2 Some QA/QC parameters may not be required for a certain method. The laboratory is often running additional QC standards beyond standard requirements. QC should meet the minimum required by the reported method

19.3 If QC biases high, and samples are below detection limits, results can be reported.

19.4 If an analyst cannot solve QA/QC problems, an irregularity report should be filled out and given to QA/QC for evaluation. If instrument problems persist, maintenance and troubleshooting should be performed to fix the problem.

20.0 Waste Management and Pollution Prevention

20 1 This procedure generates acidic wastes. All wastes shall be disposed of according to local regulatory as well as laboratory-established guidelines. Refer to the Laboratory's Disposal SOP A-2050

20.2 Pollution prevention is any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. This procedure generates acidic waste and relies on concentrated metals standards for preparation. The laboratory should minimize the amount of acids used whenever possible. Small volumes of standards are used to create calibration and QC standards. Concentrated metals standards should be ordered in quantities sufficient to be fully utilized by their expiration date, to eliminate unnecessary disposal of concentrated metals standards.

21.0 References

21.1 *Test Methods for Evaluating Solid Wastes, SW-846*, Third Edition, Update III, July 1992, Method 6010.

21.2 Sample Waste Disposal, A-2050.

21.3 *Ohio EPA Laboratory Manual for Chemical Analyses of Public Drinking Water*, 2000

21.4 *Guide to Environmental Analytical Methods*, Edited by Robert E. Wagner and William Kotas, Genium Publishing Corporation, 2nd Edition 1992

21.5 *Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*, EMMC Version, Revision 4.4, 1994

21.6 *Standard Methods for the Examination of Water and Wastewater18th Edition*, Edited by Arnold E. Greenburg, Lenore S. Clesceri, and Andrew D. Eaton, American Public Health Association, 1992

21.7 *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, Office of Superfund Remediation and Technology Innovation USEPA, OSWER 9240.1-45, EPA 540-R-04-004, October 2004

Appendix A. Drinking Water Testing Requirements

The following procedures are followed when analyzing drinking water samples for metals; these procedures are additions or modifications to normal liquid sample procedures.

(a) A reporting limit check (RLC) must be analyzed at the beginning of the analytical run. It should be analyzed immediately following the ICV and ICB.

(i) Prepared concentration should be at the reporting limit for the element of interest. Prepare from the primary source standards.

(ii) Acceptance criteria is ± 30%. If the RLC fails, prepare a second RLC and check; if the second check fails, perform an instrument recalibration.

(b) Run samples in the following order: MB, LCS, sample A, sample B, etc., including duplicate samples and MS/MSD samples at a frequency of one in ten or fewer samples. If the spike recovery or RPD is out of the acceptable range, the sample must be diluted and spiked. If it fails again, the sample must be analyzed by the methods of standard additions.

(c) After each ten analyses (spikes count as one analysis each), run a CCV and CCB. If the CCV does not meet acceptance criteria, terminate the sequence, correct the problem, recalibrate and re-analyze the samples and spikes between the last acceptable CCV and the failed CCV.

(d) When all samples and spikes are analyzed, re-analyze the CCB, CCV.

(e) At least once every three months a minimum of one actual drinking water sample must be analyzed to maintain certification. If no sample has come in, analyze a tap water sample from the lab. All certified elements must be analyzed for and documented.

Table A1: QC Acceptance Criteria for Drinking Water

Standard	Acceptance Criteria
Instrument Blank (initial)	Zero
Calibration Curve	r-value = 0.995 or greater
ICV	%Recovery = 95-105%
ICB	Below reporting limit
RLC	%Recovery = 70-130%
RS	%Recovery = 90-110%
MB	Below reporting limit
LCS	%Recovery = 85-115%
MS/MSD (above RL)	%Recovery = 80-120%
MS/MSD (below RL)	%Recovery = 70-130%
Duplicates	RPD = ≤20% if value is <25x MDL, RPD = ≤10% if value is >20x MDL
CCV	%Recovery = 90-110%
CCB	Below reporting limit

Appendix B. Lead Testing for AIHA ELLAP Certification

The following procedures are followed when analyzing lead samples requiring AIHA ELLAP certification. Samples matrices include air, wipes, paint or soil

(a) Detection limit studies must establish a reporting limit equal to or less than 20% of the lowest relevant action level action level or regulatory limit, except for lead wipes. Lead wipes should be equal to or less than 50% of the lowest relevant action level. National limits of interest are: paint = 0.06%; soil = 400 ppm; wipe = 40 ug/ft2. State regulatory limits may be lower than national limits Applicable state limits must be checked before analyzing samples

(b) MDL studies should be established for each matrix as part of method validation, and should be performed annually thereafter. If any changes occur in methodology and instrumentation, MDLs should be rechecked.

(c) An Initial Calibration Verification (ICV) standard must be analyzed after calibration and before analyzing any samples. The concentration should be near the regulatory level of concern, and be prepared from a secondary source. The ICV should be matrix matched to the acid concentration of digested samples Acceptance criteria is given in Table B1

(d) An Interference Check Solution (ICS) should be analyzed at the beginning and end of each run or twice every eight hours. The ICS should be matrix matched to the acid concentration of digested samples Acceptance criteria is given in Table B1.

(e) Reporting Limit Check (RLC): each day that a matrix is analyzed, a matrix spiked sample at a concentration equal to or below the stated reporting limit shall be digested and analyzed to demonstrate acceptable recovery of lead The acceptance criteria shall be +/- 20% of the actual value. A liquid spike is acceptable

(f) A Continuing Calibration Verification (CCV) standard should be run every 10 samples and at the end of the run The concentration should be near the midpoint of expected linearity The CCV should be matrix matched to the acid concentration of digested samples. Acceptance criteria is given in Table B1

(g) Laboratory Control Sample (LCS): Should be run once per 20 samples or less, or in batches of 5%. It must be taken from a secondary standard source, and should match the matrix of samples being analyzed. The LCS is carried through the entire procedure with the sample batch from digestion through analysis. Liquid spikes may not be used except for dust matrices.

Table B1: QC Acceptance Criteria for AIHA ELLAP

Standard	Description	Frequency	Acceptance Criteria
ICV		Once per run after calibration	%Recovery = 90-110%
ICB	Used for init. Calibration and zeroing instrument response	Once per run at the beginning of the run	Absolute value not more than 10% of regulatory limit or level of concern.
ICS (Interference Check Solution)	Conc. @ lower end of range – 5 ppm or lower if possible	Beginning and end of each run, or twice in an 8 hour period	%Recovery = 80-120%
RLC	Digested, matrix-specific, conc. @ reporting level; prepare by adding liquid standard to matrix blank sample.	Daily	%Recovery = 80-120%
MB	Digested, matrix-specific	One per 20 samples or batch	Absolute value not more than 10% of regulatory

		(5%)	limit or level of concern.
LCS/LCSD	• Digested, matrix-specific • Conc. @ mid-range: 5-10 ppm (uncorrected instrument value) • All spiking standards to be either NIST traceable or an ELPAT source: must be a like-matrix. • Wipe-prepare by adding solid dust to blank wipe. • Paint, Soil-use purchased standard. • Paint,Soil-only need LCS if dups and MS/MSD performed on client samples. • Air filter-prepare by adding liquid Std to blank filter.	One per 20 samples or batch (5%)	%Recovery = 80-120%
MS/MSD	Perform on Paint and Soil. Prepare by adding liquid Std to duplicate sample.	One per 20 samples or batch (5%)	%Recovery = 75-125%, RPD: ≤ 20%
Duplicates	Perform on Paint and Soil	One per 20 samples or batch (5%)	RPD = ≤ 20%
CCV	Conc. @ mid-range: 10 ppm	Every 10 samples and at the end of the run.	%Recovery = 90-110% for ICP
CCB	Used to verify blank responses and freedom from carryover	After each ICS and CCV	Absolute value not more than 10% of regulatory limit or level of concern.

Appendix C Recommended Wavelengths

Analyte	Primary Wavelength (nm)	Secondary Wavelength (nm)
Aluminum	394.40	308.22
Antimony	217.58	206.83
Arsenic	193.70	188.98
Barium	233.53	455.40
Beryllium	234.86	313.04
Cadmium	228.80	226.50
Calcium	317.93	315.89
Chromium	357.87	267.72
Cobalt	238.89	228.62
Copper	324.75	219.96
Iron	238.20	259.94
Lead	220.35	217.00
Magnesium	383.83	279.08
Manganese	257.61	294.92
Molybdenum	281.62	202.03
Nickel	231.60	221.65
Potassium	766.49	769.90
Selenium	196.03	203.99
Silver	328.07	338.29
Sodium	589.59	589.00
Strontium	421.55	407.77
Thallium	190.79	351.924
Tin	189.93	
Titanium	334.94	
Vanadium	292.40	
Zinc	213.86	206.20

MDL Study Metals Varian ICP 2007

Analysis Method: SW846-6010B/200.7 Analyst: RLG

Prep Method: SW846-3010A, 3005A

Metal	Matrix	Prep Date	Spiking Conc. (mg/L)	Replicate Number / Analysis Date / Results 1	2	3	4	5	6	7	Samples (N)	Deviation (s)	Calc. MDL (mg/L)	Reporting Limit (mg/L)	Solid Reporting Limit** (mg/kg)	Mean (mg/L)	Average %Rec
				2/5/2007	2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/9/2007							
Ag	Liquid	2/5/2007	0.0125	0.017791	0.016215	0.010649	0.014389	0.014576	0.016889	0.011368	7	0.0027116	0.00852	0.01	0.5	0.0146	116.4%
				2/5/2007	2/5/2007	2/5/2007	2/19/2007	2/19/2007	2/19/2007	2/22/2007							
Al	Liquid	2/5/2007	0.10	0.1046	0.11823	0.10461	0.11175	0.11359	0.1114	0.12578	7	0.0074828	0.02352	0.05	2.5	0.1129	112.9%
				2/19/2007	2/19/2007	2/19/2007	3/1/2007	3/1/2007	3/1/2007	3/1/2007							
As (188)	Liquid	2/5/2007	1.00	1.1025	1.0549	1.1087	1.1033	1.0922	1.1239	1.142	7	0.0271258	0.08526	0.1	5	1.1039	110.4%
				2/22/2007	2/22/2007	2/22/2007	3/1/2007	3/1/2007	3/1/2007	3/1/2007							
As (193)	Liquid	2/5/2007	1.00	1.1031	1.0461	1.11	1.0958	1.0953	1.0827	1.1002	7	0.0212776	0.06688	0.1	5	1.0905	109.0%
				2/19/2007	2/19/2007	2/19/2007	2/22/2007	2/26/2007	2/26/2007	2/26							
Ba	Liquid	2/5/2007	0.10	0.11123	0.10974	0.10819	0.11253	0.11282	0.11165	0.11465	7	0.0021159	0.00665	0.01	0.5	0.1115	111.5%
				2/6/2007	2/6/2007	2/6/2007	2/9/2007	2/9/2007	2/9/2007	2/13/2007							
Be	Liquid	2/5/2007	0.005	0.005197	0.005114	0.00564	0.005102	0.005301	0.005335	0.005069	7	0.0001991	0.00063	0.01	0.5	0.0053	105.0%
				2/6/2007	2/6/2007	2/6/2007	2/19/2007	2/19/2007	2/19/2007	3/2/2007							
Ca	Liquid	2/5/2007	0.50	0.58939	0.56528	0.52627	0.54539	0.55096	0.56404	0.56865	7	0.0200056	0.06288	0.10	5	0.5586	111.7%
				2/5/2007	2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/9/2007							
Cd	Liquid	2/5/2007	0.025	0.026555	0.024531	0.02521	0.028199	0.028027	0.026625	0.025377	7	0.0014085	0.00443	0.01	0.5	0.0264	105.4%
				2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/19/2007	2/19/2007	2/19/2007							
Co	Liquid	2/5/2007	0.025	0.026539	0.025349	0.029831	0.029974	0.024688	0.023683	0.02828	7	0.0025074	0.00788	0.01	0.5	0.0269	107.6%
				2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/9/2007	2/9/2007	2/9/2007							
Cr	Liquid	2/5/2007	0.0125	0.017259	0.013002	0.012361	0.014584	0.013924	0.01162	0.014087	7	0.0018316	0.00576	0.01	0.5	0.0138	110.7%
				2/5/2007	2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/9/2007							
Cu	Liquid	2/5/2007	0.0125	0.014199	0.012371	0.012931	0.010452	0.012945	0.010974	0.018491	7	0.0026559	0.00835	0.01	0.5	0.0132	105.6%
				2/5/2007	2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/13/2007							
Fe	Liquid	2/5/2007	0.05	0.048144	0.057485	0.052141	0.057242	0.055282	0.056979	0.047972	7	0.0042066	0.01322	0.02	1	0.0536	107.2%
				2/5/2007	2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/13/2007							
K	Liquid	2/5/2007	0.05	0.052931	0.047811	0.051534	0.046793	0.044157	0.047173	0.050177	7	0.0030413	0.00956	0.05	2.5	0.0487	97.3%
				2/5/2007	2/5/2007	2/5/2007	2/6/2007	2/26/2007	2/26/2007	2/26/2007							
Mg	Liquid	2/5/2007	0.50	0.37199	0.37829	0.39577	0.35782	0.36838	0.37583	0.38255	7	0.0118562	0.03726	0.05	2.5	0.3758	75.2%
				2/5/2007	2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/9/2007							
Mn	Liquid	2/5/2007	0.025	0.026715	0.026142	0.025997	0.029931	0.029549	0.028397	0.029544	7	0.0017212	0.00541	0.01	0.5	0.0280	112.2%
				2/5/2007	2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/9/2007							
Mo	Liquid	2/5/2007	0.05	0.049453	0.047043	0.043455	0.055952	0.051253	0.048027	0.048985	7	0.0038501	0.01210	0.02	1	0.0492	98.3%
				2/7/2007	2/7/2007	2/7/2007	2/9/2007	2/9/2007	2/9/2007	2/13/2007							
Na	Liquid	2/5/2007	0.50	0.47409	0.48034	0.48283	0.45962	0.4533	0.46298	0.48281	7	0.0121324	0.03813	0.05	2.5	0.4709	94.2%
				2/5/2007	2/5/2007	2/19/2007	2/19/2007	2/19/2007	2/26/2007	3/2/2007							
Ni	Liquid	2/5/2007	0.025	0.026709	0.030757	0.024335	0.029944	0.026356	0.028872	0.029797	7	0.0023483	0.00738	0.01	0.5	0.0281	112.4%
				2/5/2007	2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/9/2007							
Pb	Liquid	2/5/2007	0.05	0.043016	0.055833	0.048002	0.04551	0.052379	0.049199	0.052379	7	0.0044154	0.01388	0.02	1	0.0495	98.9%
				2/9/2007	2/19/2007	2/19/2007	2/19/2007	2/22/2007	2/22/2007	2/22/2007							
Sb	Liquid	2/5/2007	0.50	0.44284	0.49511	0.52207	0.49771	0.52659	0.52995	0.51216	7	0.0300988	0.09460	0.1	5	0.5038	100.8%
				2/19/2007	2/19/2007	2/19/2007	2/22/2007	2/22/2007	2/22/2007	2/26/2007							
Se (196)	Liquid	2/5/2007	1.00	1.1987	1.2087	1.2003	1.1428	1.1556	1.1585	1.158	7	0.0268052	0.08425	0.1	5	1.1747	117.5%
				2/19/2007	2/19/2007	2/19/2007	2/22/2007	2/22/2007	2/22/2007	2/26/2007							
Se (203)	Liquid	2/5/2007	1.00	1.1605	1.1452	1.1729	1.1386	1.1746	1.2064	1.155	7	0.0226470	0.07118	0.1	5	1.1647	116.5%
				2/19/2007	2/19/2007	2/19/2007	2/22/2007	2/22/2007	2/22/2007	2/27/2007							
Sn	Liquid	2/5/2007	1.00	1.1117	1.1308	1.1479	1.1045	1.1108	1.1313	1.1098	7	0.0158848	0.04993	0.1	5	1.1210	112.1%
				2/5/2007	2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/9/2007							

MDL Study Metals Varian ICP 2007

Analysis Method: SW846-6010B/200.7 **Analyst: RLG**

Prep Method: SW846-3010A, 3005A

Metal	Matrix	Prep Date	Spiking Conc. (mg/L)	Replicate Number / Analysis Date / Results 1	2	3	4	5	6	7	Samples (N)	Deviation (s)	Calc. MDL (mg/L)	Reporting Limit (mg/L)	Solid Reporting Limit** (mg/kg)	Mean (mg/L)	Average %Rec
Sr	Liquid	2/5/2007	0.05	0.057339	0.057291	0.057256	0.059833	0.058196	0.059251	0.055442	7	0.0014562	0.00458	0.01	0.5	0.0578	115.6%
				2/19/2007	2/19/2007	2/19/2007	2/22/2007	2/22/2007	2/27/2007	2/27/2007							
Tl (352)	Liquid	2/5/2007	1.00	1.1087	1.0299	1.0672	1.1222	1.0692	1.0504	1.0958	7	0.0328670	0.10330	0.1	5	1.0776	107.8%
				2/19/2007	2/19/2007	2/19/2007	2/22/2007	2/22/2007	2/27/2007	2/27/2007							
Tl (191)	Liquid	2/5/2007	1.00	1.0918	1.1563	1.1288	1.1202	1.1005	1.1322	1.1422	7	0.0226055	0.07105	0.1	5	1.1246	112.5%
				2/5/2007	2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/13/2007							
V	Liquid	2/5/2007	0.025	0.018336	0.02196	0.01576	0.023332	0.01733	0.019859	0.015481	7	0.0030069	0.00945	0.01	0.5	0.0189	75.5%
				2/5/2007	2/5/2007	2/5/2007	2/7/2007	2/7/2007	2/7/2007	2/9/2007							
Zn (213)	Liquid	2/5/2007	0.025	0.028828	0.028829	0.029589	0.03361	0.030582	0.031056	0.029314	7	0.0017031	0.00535	0.01	0.5	0.0303	121.0%

All metals results were taken from separately prepared, separately digested replicates

****Soil Reporting limits are calculated based on the liquid reporting limit using standard solid digestion preparation (1.00 g to 50 mL)**

2007 LCS Control Data for Varian ICP

																											Replicates	SD	Mean	Average % Rec
	6/1/2007	6/1/2007	6/1/2007	6/1/2007	5/29/2007	5/29/2007	5/25/2007	5/25/2007	5/23/2007	5/23/2007	5/9/2007	5/9/2007	4/5/2007	4/5/2007	3/6/2007	3/6/2007														
Al	1.091	1.0968	1.0614	1.0908	1.0506	1.083	1.0359	1.0321	0.90264	1.072	1.0146	1.0331	1.0294	0.99297	1.1302	1.1659											16	0.06007181	1.05515063	106%
	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/18/2007	5/18/2007	5/17/2007	5/17/2007	5/17/2007	5/17/2007	5/16/2007	5/16/2007	5/15/2007	5/15/2007	5/14/2007	5/14/2007	5/14/2007	5/14/2007	5/11/2007	5/11/2007	5/11/2007	5/11/2007	5/9/2007					
As (193)	0.9602	0.9938	1.108	1.0487	0.99277	1.1085	1.1718	1.0311	1.0824	1.0227	0.99931	1.0785	1.0235	1.0515	1.1085	1.0585	0.9102	1.1334	1.0185	0.9199	1.108	1.1039	1.0414	0.94741	1.0113		25	0.06669816	1.0413516	104%
	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/18/2007	5/18/2007	5/17/2007	5/17/2007	5/17/2007	5/17/2007	5/16/2007	5/16/2007	5/15/2007	5/15/2007	5/14/2007	5/14/2007	5/14/2007	5/14/2007	5/11/2007	5/11/2007	5/11/2007	5/11/2007	5/9/2007					
As (188)	1.0699	1.1103	1.1533	1.0001	1.1956	1.0219	1.0549	1.0559	1.0506	1.1376	0.98169	1.1255	1.2353	1.0456	1.0631	1.1359	1.0103	0.95116	1.2391	1.2925	1.0423	1.1494	0.90285	0.83279	1.2586		25	0.11131286	1.0846476	108%
	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/18/2007	5/18/2007	5/17/2007	5/17/2007	5/17/2007	5/17/2007	5/16/2007	5/16/2007	5/15/2007	5/15/2007	5/14/2007	5/14/2007	5/14/2007	5/14/2007	5/11/2007	5/11/2007	5/11/2007	5/11/2007	5/8/2007					
Ba	1.1174	1.1213	0.98415	0.98069	1.1159	1.1157	1.0655	1.108	1.0187	1.0138	1.0599	1.0846	1.1023	1.1094	1.0965	1.0734	1.0316	1.0525	1.0228	1.041	0.98093	0.99312	0.98737	0.97215	0.9797		25	0.05350453	1.0499364	105%
	6/1/2007	6/1/2007	6/1/2007	6/1/2007	6/1/2007	5/31/2007	5/31/2007	5/30/2007	5/30/2007	5/25/2007	5/25/2007	5/17/2007	5/17/2007	5/14/2007	5/14/2007	4/20/2007	4/20/2007	4/20/2007	4/20/2007	4/19/2007	4/19/2007	3/13/2007	3/13/2007	2/20/2007	2/20/2007					
Be	1.105	1.106	1.0656	1.0869	1.1038	1.1137	1.1108	1.0435	1.0442	1.0498	1.0555	1.0695	1.0726	1.0197	1.0203	1.0422	1.0665	1.1474	1.1299	1.0635	1.1443	1.0393	1.0506	1.1151	1.1099		25	0.03728928	1.079024	108%
	6/1/2007	5/17/2007	5/17/2007	5/17/2007	5/17/2007	4/9/2007	4/9/2007	3/30/2007	3/30/2007	3/16/2007	3/16/2007	3/14/2007	3/14/2007	3/2/2007	3/2/2007	3/2/2007	3/2/2007	3/2/2007	3/2/2007	2/22/2007	2/22/2007	2/19/2007	2/19/2007	2/20/2007	2/20/2007					
Ca	1.133	1.0821	1.0865	1.1333	1.1388	1.2598	1.2547	1.1582	1.1469	1.1588	1.1595	1.0175	1.057	1.0191	1.0117	1.3649	1.268	0.99953	1.0175	1.249	1.1662	1.118	1.1105	1.0014	1.0065		25	0.0988972	1.1247372	112%
	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/18/2007	5/18/2007	5/18/2007	5/18/2007	5/17/2007	5/17/2007	5/17/2007	5/17/2007	5/16/2007	5/16/2007	5/15/2007	5/15/2007	5/14/2007	5/14/2007	5/14/2007	5/14/2007	5/11/2007	5/11/2007	5/10/2007					
Cd	1.1047	1.101	0.96161	0.95845	1.0931	1.1086	1.1184	1.1169	1.0798	1.0812	1.0115	1.027	1.0663	1.0613	1.0772	1.0841	1.0778	1.0885	1.0815	1.0858	1.0449	1.0889	1.1085	1.0823	1.0071		25	0.04381426	1.0686584	107%
	6/1/2007	6/1/2007	6/1/2007	6/1/2007	5/4/2007	5/4/2007	4/20/2007	4/20/2007	3/20/2007	3/20/2007																				
Co	1.1193	1.1233	1.079	1.1153	1.0728	1.0344	1.0502	1.0673	1.1199	1.133																	10	0.03486001	1.09145	109%
	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/18/2007	5/18/2007	5/18/2007	5/18/2007	5/17/2007	5/17/2007	5/17/2007	5/17/2007	5/16/2007	5/16/2007	5/16/2007	5/16/2007	5/15/2007	5/15/2007	5/14/2007	5/14/2007	5/14/2007	5/14/2007	5/11/2007					
Cr	1.0212	1.0177	0.91852	0.93389	1.0479	0.96216	1.0054	1.01	1.0364	0.97325	0.94191	0.93715	0.99554	1.0613	1.0131	0.95111	0.86545	0.80753	0.96203	0.95524	0.93019	0.95737	0.89979	1.0265	0.93375		25	0.05909719	0.9665752	97%
	5/18/2007	5/18/2007	5/18/2007	5/18/2007	5/17/2007	5/17/2007	5/16/2007	5/16/2007	5/10/2007	5/10/2007	5/10/2007	5/10/2007	5/9/2007	5/9/2007	5/8/2007	5/8/2007	5/2/2007	5/2/2007	4/30/2007	4/30/2007	4/26/2007	4/26/2007	4/25/2007	4/25/2007	4/24/2007					
Cu	1.0657	0.98503	1.0701	1.0698	1.0657	1.061	1.0704	1.0613	0.99564	0.99212	1.0406	0.99506	1.0739	1.063	0.97585	0.97375	0.96564	1.0521	1.0286	1.0303	1.1102	1.0775	1.119	0.99236	1.0588		25	0.04354825	1.039738	104%
	5/18/2007	5/18/2007	5/17/2007	5/17/2007	5/17/2007	5/17/2007	5/9/2007	5/9/2007	5/9/2007	5/9/2007	4/26/2007	4/26/2007	4/20/2007	4/20/2007	4/5/2007	4/5/2007	4/3/2007	4/3/2007	4/2/2007	4/2/2007	3/22/2007	3/22/2007	3/8/2007	3/8/2007	3/6/2007					
Fe	1.1495	1.1257	1.112	1.111	1.0884	1.0886	1.1443	1.0764	1.0553	1.0381	1.0501	1.0567	1.054	1.0604	1.0979	1.0758	1.0915	1.0401	1.075	1.0871	1.1042	1.1067	1.1023	1.1265	1.1153		25	0.0311118	1.089316	109%
	6/1/2007	6/1/2007	6/1/2007	6/1/2007	5/17/2007	5/17/2007	5/17/2007	4/5/2007	4/5/2007	3/22/2007	3/22/2007	3/16/2007	3/16/2007																	
K	8.214	8.1715	8.3692	8.5775	8.1587	8.405	8.1123	8.8288	8.5522	9.4627	8.7934	8.5788	8.4745														13	0.36699	8.51527692	85%
	6/1/2007	6/1/2007	6/1/2007	6/1/2007	5/17/2007	5/17/2007	5/17/2007	5/17/2007	3/30/2007	3/30/2007	3/16/2007	3/16/2007	3/14/2007	3/14/2007	3/2/2007	3/2/2007	3/2/2007	3/2/2007	3/2/2007	3/2/2007	3/2/2007	2/20/2007	2/20/2007							
Mg	1.1081	1.1168	1.1306	1.1086	1.077	1.0889	1.0984	1.1272	1.0872	1.0496	1.2077	1.2169	1.0146	1.0675	1.0364	1.0542	1.0892	1.099	0.82717	1.0572	1.0617	1.105	1.0725				23	0.0729078	1.08267261	108%
	6/1/2007	6/1/2007	6/1/2007	6/1/2007	5/9/2007	5/9/2007	4/26/2007	4/26/2007	4/20/2007	4/20/2007	4/5/2007	4/5/2007	3/8/2007	3/8/2007	3/6/2007	3/6/2007	2/20/2007	2/20/2007												
Mn	1.1204	1.0951	1.1018	1.1164	1.1426	1.113	1.0334	1.0461	1.058	1.0755	1.1208	1.0756	1.0957	1.0762	1.0478	1.1062	1.0098	1.0094									18	0.03916253	1.08021111	108%
	6/4/2007	6/4/2007	6/1/2007	6/1/2007	6/1/2007	6/1/2007	5/30/2007	5/30/2007	5/18/2007	5/18/2007	5/18/2007	5/18/2007	5/17/2007	5/17/2007	5/14/2007	5/14/2007	5/9/2007	5/9/2007	4/26/2007	4/26/2007	3/29/2007	3/29/2007	3/7/2007	3/7/2007						
Mo	0.90598	0.90323	1.0375	1.0423	1.039	1.0208	1.0213	0.97342	0.98053	0.90846	1.0178	1.0424	1.0684	1.0655	1.0084	0.98732	0.99456	0.96812	0.92557	0.91648	0.97443	0.96266	1.0055	0.96857			24	0.05037225	0.98909292	99%
	6/1/2007	6/1/2007	6/1/2007	6/1/2007	4/5/2007	4/5/2007	3/22/2007	3/22/2007	3/16/2007	3/16/2007	3/2/2007	3/2/2007	3/2/2007	3/2/2007	3/2/2007	3/2/2007	3/2/2007	2/19/2007	2/19/2007											
Na	0.8336	0.8338	0.88508	0.87708	1.1522	1.1546	0.97147	0.94294	1.0327	1.0395	0.82064	0.83151	0.94707	0.93775	1.1489	0.82625	0.82662	0.87091	0.89688								19	0.11600721	0.93839474	94%
	5/22/2007	5/22/2007	5/18/2007	5/18/2007	5/18/2007	5/18/2007	5/16/2007	5/16/2007	5/10/2007	5/10/2007	5/10/2007	5/10/2007	5/9/2007	5/9/2007	5/9/2007	5/9/2007	5/8/2007	5/8/2007	5/2/2007	5/2/2007	4/30/2007	4/30/2007	4/26/2007	4/26/2007	4/25/2007					
Ni	1.0865	1.0506	1.0725	1.0466	1.1378	1.0705	1.1226	1.1095	1.0162	1.0119	1.0474	0.99468	1.0882	1.0875	1.0402	1.0355	0.98844	0.98154	1.0193	1.124	1.0485	1.0626	1.113	1.094	1.1636		25	0.04839674	1.0645264	106%
	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/18/2007	5/18/2007	5/18/2007	5/18/2007	5/17/2007	5/17/2007	5/17/2007	5/17/2007	5/16/2007	5/16/2007	5/15/2007	5/15/2007	5/15/2007	5/15/2007	5/15/2007	5/15/2007	5/14/2007	5/14/2007	5/11/2007					
Pb	1.1116	1.1301	1.0105	1.0037	1.1603	1.1698	1.1305	1.0969	1.1335	1.133	1.0537	1.0604	1.1103	1.1233	1.1689	1.1324	1.0892	1.0726	1.1133	1.1256	1.1246	1.1386	1.0571	1.0603	0.96212		25	0.0525726	1.0988928	110%
	5/17/2007	5/17/2007	5/14/2007	5/14/2007	5/1/2007	5/1/2007	5/1/2007	5/1/2007	4/30/2007	4/30/2007	4/30/2007	4/30/2007	4/20/2007	4/20/2007	4/20/2007	4/20/2007	4/19/2007	4/19/2007	4/18/2007	4/18/2007	4/18/2007	4/18/2007	3/13/2007	3/13/2007	2/20/2007					
Sb	0.87246	0.86167	1.0756	1.068	1.1577	1.1685	0.86144	1.1106	1.3184	1.3175	1.1458	1.0315	1.0667	1.0497	1.0812	1.0416	1.1043	1.1463	0.96349	0.94283	1.0066	0.9763	1.0532	1.0498	1.1203		25	0.11591057	1.0644596	106%
	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/18/2007	5/18/2007	5/17/2007	5/17/2007	5/16/2007	5/16/2007	5/15/2007	5/15/2007	5/14/2007	5/14/2007	5/11/2007	5/11/2007	5/10/2007	5/10/2007	5/10/2007	5/10/2007	5/8/2007	5/8/2007	5/4/2007					
Se (196)	1.091	1.1179	1.3453	1.2617	1.3495	1.3944	1.1342	1.7493	1.1607	1.1918	1.1687	1.27	1.1653	1.1513	1.2529	1.0912	1.1399	1.1209	0.99473	1.1451	1.0788	1.1211	1.2848	1.2955	1.0649		25	0.1507132	1.2056372	121%
	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/22/2007	5/18/2007	5/18/2007	5/17/2007	5/17/2007	5/16/2007	5/16/2007	5/15/2007	5/15/2007	5/14/2007	5/14/2007	5/11/2007	5/11/2007	5/10/2007	5/10/2007	5/10/2007	5/10/2007	5/8/2007	5/8/2007	5/4/2007					
Se (203)	1.783	1.5514	1.9673	1.7809	1.6028	1.5319	1.1892	1.4606	1.1918	1.318	1.5571	1.1656	1.3983	1.2965	1.3411	1.5537	1.0363	1.3607	1.3028	1.1481	1.2762	1.6807	1.3227	1.4673	1.4133		25	0.22291906	1.428692	143%
	6/5/2007	6/5/2007	5/14/2007	5/14/2007	5/9/2007	5/9/2007																								
Sn	1.1228	1.1112	1.03	0.97537	1.0046	1.0786																					6	0.05968105	1.05376167	105%
	6/1/2007	6/1/2007	5/9/2007	5/9/2007	4/3/2007	4/3/2007	3/7/2007	3/7/2007																						
Sr	1.1716	1.1761	1.1173	1.1105	1.1476	1.1106	1.0741	1.0604																			8	0.04210191	1.121025	112%
	6/1/2007	5/31/2007	5/31/2007	5/30/2007	5/30/2007	5/29/2007	5/29/2007	5/25/2007	5/25/2007	5/17/2007	5/17/2007	5/14/2007	5/14/2007	4/20/2007	4/20/2007	4/20/2007	4/20/2007	4/19/2007	4/19/2007	3/13/2007	3/13/2007	2/28/2007	2/28/2007	2/20/2007	2/20/2007					
Tl (191)	1.1276	1.0414	1.0983	1.1612	0.9203	0.95658	0.91897	1.1429	1.0768	1.0711	1.2714	1.0678	1.0449	1.0555	0.86919	1.0634	1.0319	1.0816	1.2041	1.1957	1.165	1.1323	1.1606	1.2199	1.2455		25	0.10308846	1.0929576	109%
	6/1/2007	5/31/2007	5/31/2007	5/30/2007	5/30/2007	5/29/2007	5/29/2007	5/25/2007	5/25/2007	5/17/2007	5/17/2007	5/14/2007	5/14/2007	4/20/2007	4/20/2007	4/20/2007	4/20/2007	4/19/2007	4/19/2007	3/13/2007	3/13/2007	2/28/2007	2/28/2007	2/20/2007	2/20/2007					
Tl (352)	1.1432	1.0056	1.0376	0.9418	1.0489	1.0253	1.1555	0.96146	1.186	1.1124	1.0287	1.08	1.0105	0.99838	0.99665	0.8981	0.8773	1.1174	1.0835	0.85288	0.92584	1.0881	0.98498	1.0967	1.1382		25	0.08970041	1.0317996	103%
	6/1/2007	6/1/2007	6/1/2007	6/1/2007	4/20/2007	4/20/2007	3/6/2007	3/6/2007	2/21/2007	2/21/2007																				
V	1.0377	1.0465	1.009	1.0334	0.97714	0.98246	1.0146	1.0056	0.98911	0.98277																	10	0.02507221	1.007828	101%
	5/22/2007	5/22/2007	5/18/2007	5/18/2007	5/18/2007	5/18/2007	5/16/2007	5/16/2007	5/14/2007	5/14/2007	5/10/2007	5/10/2007	5/10/2007	5/10/2007	5/9/2007	5/9/2007	5/9/2007	5/9/2007	5/8/2007	5/8/2007	5/3/2007	5/3/2007	5/2/2007	5/2/2007	5/1/2007					
Zn (213)	1.0571	1.0691	1.0689	1.1261	1.1434	1.1349	1.093	1.0902	0.99085	0.97493	1.0015	1.0109	1.0796	1.0644	1.1458	1.1356	1.0726	1.0544	1.003	1.0067	1.1197	1.1126	0.9868	1.0913	1.1186		25	0.05411464	1.0700792	107%
	5/22/2007	5/22/2007	5/18/2007	5/18/2007	5/18/2007	5/18/2007	5/16/2007	5/16/2007	5/14/2007	5/14/2007	5/10/2007	5/10/2007	5/10/2007	5/10/2007	5/9/2007	5/9/2007	5/9/2007	5/9/2007	5/8/2007	5/8/2007	5/3/2007	5/3/2007	5/2/2007	5/2/2007	5/1/2007					
Zn (206)	1.1009	1.1081	1.0589	1.0671	1.246	1.2329	1.1151	1.1428	0.99371	0.97135	0.97697	0.97161	1.0745	1.0272	1.1701	1.1764	1.057	1.0491	1.0402	1.0434	1.126	1.1315	1.0052	1.1131	1.102		25	0.0752033	1.0840456	108%

FIRSTECHNOLOGY, Inc.

Standard Operating Procedures

SOP No.: SOP/M/1600

Mercury by Manual Cold Vapor Atomic Absorption

Approved By: _____, **Lab Manager**
Mark Kalmeyer

Approved By: _____, **QA/QC Officer**
Scott Bolam

Revision: 3

SOP Effective Date: 6/4/07

Controlled Copy on Green Paper

Copy No. _____
Approved By _____
Date _____

1.0 Methods

1.1. This SOP meets the requirements of the following methods: 245.1, SM3112, SW7471, and SW7470.

2.0 Applicable Matrices

2.1 This method applies to a wide range of environmental matrices including drinking, surface, and saline waters, mobility extracts, soils, sludges, sediments, oils and domestic and industrial waste water.

3.0 Detection Limits

3.1 The reporting limits for mercury are 0.2 ug/L for water, 0.5 ug/L for TCLP extracts, and 20 ug/Kg in soils.

4.0 Scope and Application

4.1 This SOP encompasses measuring total mercury in drinking water, surface and ground water, industrial, and domestic waste water, as well as soils, sludges, sediments, and oils.

4.2 The method cited is dependent on the sample matrix. Methods are assigned to samples during login. See section 1.0 for a list of methods covered by this SOP.

4.3 The standard working range for aqueous samples is 0.2 to 10 ug/L. This range may be adjusted based on sample matrix and operating conditions.

4.4 For drinking water analysis, see Appendix A for additions/modifications to the normal procedures outlined in this SOP.

5.0 Summary

5.1 Prior to analysis by cold vapor generation, samples must be prepared in order to convert organic forms of mercury to inorganic forms, to minimize organic interferences, and to convert the sample to a suitable solution for analysis. The sample preparation procedure varies depending on the sample matrix.

5.2 Dissolved Mercury can be analyzed directly provided the sample is filtered with a 0.45 µm filter prior to acid preservation. Filtered samples may be analyzed directly according to the procedures contained in this SOP. Mercury, however, must always undergo the permanganate digestion procedure for liquids. The acid concentration in the samples must be the same as the acid concentration in the calibration standards.

5.3 A light beam specific for mercury (wavelength of 253.7) from a hollow cathode lamp or an electrodeless discharge lamp (EDL) is directed through a quartz cell located in the optical path of an atomic absorption spectrophotometer into the monochromator and on to a detector which measures the absorbed radiation of the aerated mercury. The resulting absorption of the lamp radiation is proportional to the mercury concentration.

6.0 Responsibilities

6.1 Analyst: Personnel who are trained and proficient in the method

7.0 Definitions

7.1 AA: Atomic absorption spectrometer

7.2 HCL: Hollow cathode lamp

7.3 CAL STD: Calibration standard, a solution containing known concentrations of the analytes to be analyzed. A series of CAL STDs are used to calibrate the instrument.

7.4 Calibration blank, reagent-grade water and instrumental grade acids combined in the same concentrations as in the standards. It is used to perform the initial calibration of an instrument.

7.5 MB: Method blank, an aliquot of reagent water that is treated exactly like a sample. It is used to determine if method analytes or other interferences are present.

7.6 ICB: Initial calibration blank, DI water analyzed as a sample following the calibration. It must be below the reporting limit.

7.7 ICV: Initial calibration verification, a primary source standard solution with a concentration at or near the midpoint of the working calibration range. It is used to determine the validity of the calibration curve.

7.8 RS: Reference standard, a secondary source standard solution (undigested) containing a known concentration of the analytes to be analyzed. It is prepared from a different source (see secondary source) and in a different concentration than the **CAL STD**.

7.9 CCV: Continuing calibration verification, a primary source solution with a concentration at or near the midpoint of the working calibration range. It is run *after* a batch of ten or fewer samples. It must be below the reporting limit and used to determine if the calibration curve is still valid.

7.10 CCB: Continuing calibration verification, DI water analyzed *after* a batch of ten or fewer samples; it is used to determine if the calibration curve is still valid.

7.11 RLC: Reporting limit check (also called the detection limit check), a standard prepared at a concentration equal to or less than the reporting limit. It is run after the calibration.

7.12 LDR: Linear dynamic range, the concentration range over which the analytical curve remains linear.

7.13 MB: Method blank, an aliquot of reagent water that is treated exactly like a sample. It is prepared and digested with each group of twenty or fewer samples digested. It must be below the reporting limit and is used to determine if method analytes or other interferences are present.

7.14 LCS: Laboratory control sample, an aliquot of reagent water to which is added a known amount of method analytes. This is treated exactly like a sample. It is prepared and digested (usually is made from the same source as the **CAL STD**) with each group of twenty or fewer samples digested. The purpose of the LCS is to demonstrate the performance of the overall analytical procedure, including sample preparation and instrumental analysis, in a clean matrix.

7.15 DUP: Duplicate, an inter- or intralaboratory split sample, for which both aliquots are brought through the entire digestion and analysis procedure.

7.16 MDL: Method Detection Limit.

7.17 MS/MSD: Matrix spike and matrix spike duplicate, an aliquot of a selected sample to which a known amount of method analyte(s) are added. These are treated exactly like samples and are used to determine the presence of matrix interference. The spiking solution usually is made from the same source as the **CAL STD;** the MS/MSD pair is run at a frequency of one in twenty or fewer samples.

7.18 VGA: Varian VGA-76 Vapor Generation Assembly.

7.19 Secondary Source: a source of standards used that is independent of the source used for calibration. This should be from a different vendor unless it can be determined that the vendor used different standard lots to prepare the standards. Different assigned vendor lot numbers are not sufficient to determine true independence. Consult traceability documentation or contact the manufacturer.

8.0 Interferences

8.1 Potassium permanganate is added to eliminate possible interferences 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.

8.2 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.

8.3 If a sample is suspected to contain certain volatile organic materials that will interfere with the analysis, a preliminary run without reagents will be performed to determine if this type of interference is present.

8.4 Sample preparation, digestion, and analysis may be subject to environmental contamination if performed in areas with high ambient background levels where mercury may have been used as a reagent. Tests such as COD and TKN employ mercury reagents, and care should be taken to separate prep areas and equipment.

9.0 Environmental Health and Safety

9.1 Mercury hydride is extremely toxic, all digestion procedures and analysis must be preformed in a fume hood.

9.2 Laboratory coats, gloves, and safety glasses shall be worn in order to avoid exposure to strong acids and reducing agents used in this procedure.

10.0 Equipment and Supplies

10.1 Environmental Express Hot Block.

10.2 A continuous-flow hydride generator: A commercially available continuous-flow sodium borohydride/HCl hydride generator. A Varian VGA-76 Vapor Generation Assembly is used for this analysis.

10.2.1 Peristaltic Pump: A three-channel, variable-speed peristaltic pump to permit regulation of liquid-stream.

10.2.2 Transfer Tubing and Connectors: Transfer tubing (1 mm I.D.), mixing T's, and connectors are made of a fluorocarbon (PFA or TFM) and are of compatible sizes to form tight, leak-proof connections.

10.2.3 Mixing Coil: A 20-turn coil made by wrapping transfer tubing around a 1-cm diameter by 5-cm long plastic or glass rod.

10.2.4 Gas-Liquid Separator: A glass apparatus for collecting and separating liquid and gaseous products which allows the liquid fraction to drain to waste and gaseous products above the liquid to be swept by a regulated carrier gas (argon) out of the cell for analysis. To avoid undue carrier gas dilution, the gas volume above the liquid should not exceed 20 mL.

10.2.5 Flow Meter/Regulator: A meter capable of regulating up to 1 L/min of nitrogen carrier gas is recommended.

10.3 Absorbance Cell: Standard spectrophotometer cells 10 cm long with quartz end windows may be used. Suitable cells (Varian Model UGA-76 accessory or equivalent) may be constructed from Plexiglas tubing, 1 inch O.D. x 4.5 inches. The ends are ground perpendicular to the longitudinal axis, and quartz windows (1 in. diameter x 1/16 in. thickness) are cemented in place. The cell is strapped to a burner head in place and aligned according to instrument recommendations. The cell is then positioned horizontally and vertically to give the maximum transmittance.

10.4 Atomic Absorption Spectrophotometer, single or dual channel instrument with grating monochrometer, photomultiplier detector, adjustable slits, a wavelength range from 190 to 800 nm, and provision for interfacing with a graphical display or other data collection system. Instrument must have background correction capability. Consult instrument manufacturer's recommended setup and operating procedures. FIRSTECHNOLOGY, Inc utilizes a Varian SpectrAA 20 Plus.

10.5 Burner with an appropriate mounting bracket attached to the burner that suspends the quartz absorbance cell between 1 and 2 cm above the burner slot is required.

10.6 Mercury hollow cathode lamps or electrodeless discharge lamps and power supply. Super-charged hollow-cathode lamps or EDL lamps are recommended for maximum sensitivity.

10.7 Pipettes, Adjustable, Microliter with disposable tips. Sizes can range from 5 to 5,000 µL, as required. Pipette tips used should be suitable for trace metal analysis.

10.8 Volumetric Flasks, Class A, assorted sizes including 100 and 1000 mL.

10.9 Volumetric pipettes, Class A, assorted sizes from 0.5 to 5 mL.

10.10 Digestion tubes.

10.11 Graduated cylinders, Class A, various sizes from 10 to 1000 mL.

10.12 Acid dispensing bottles, polyethylene, polypropylene or equivalent material, in various dispensing sizes (1, 2, 5, and 10 mL recommended).

10.13 Heating plate, capable of maintaining a temperature of 95 ± 2°C

10.14 Quantitative filter paper, Whatman 41 or equivalent

10.15 Balance

10.16 Exhaust hood to vent toxic mercury vapor from lab area.

11.0 Reagents and Standards

All reagents must contain <MDL of mercury.

11.1 ASTM Type I reagent water.

11.2 Nitric acid (concentrated): Acid must be analyzed to determine levels of impurities.

11.3 Hydrochloric acid (concentrated): Acid must be analyzed to determine levels of impurities

11.4 Sulfuric acid (concentrated): Acid must be analyzed to determine levels of impurities

11.5 Sodium chloride/hydroxylamine sulfate solution: Dissolve 12g of sodium chloride and 12g of hydroxylamine sulfate into 100mL of reagent water Agitation and slight heating may be required to effect dissolution

11.6 Potassium permanganate, certified mercury free, 5% solution (w/v): Dissolve 5g in 100 mL of reagent water.

11.7 Potassium persulfate, 5% solution (w/v): Dissolve 5g of potassium persulfate in 100 mL of reagent water

11.8 $SnCl_2$ Channel (25% $SnCl_2$): In a 200mL volumetric flask, add 50g reagent-grade $SnCl_2$ and 100mL 1M HCl Fill to mark with 1M HCl Agitation and slight heating may be required to effect dissolution

11.9 Nitrogen (UHP grade): 200-300 cf compressed gas cylinder.

11.10 Calibration Standards and QC samples:

11.10.1 Primary source standards shall be used to prepare the initial calibration curve standards and continuing calibration verification (CCV) samples A calibration blank and a minimum of five calibration standards shall be prepared daily.

11.10.2 The primary source standard concentration is 1000 mg/L of mercury. A 1.0 mL aliquot of standard is diluted to 100mL in a volumetric flask with 5 mL conc. HNO_3 and the remainder with reagent-grade water to produce a 10.0 mg/L initial standard solution A 0.1 mg/L working standard then is prepared by diluting a 1.0 mL aliquot of the initial standard solution to 100 mL in a volumetric flask with 5 mL conc. HNO_3 and the remainder with reagent-grade water The calibration standards, ICV, CCV, LCS and MS/MSD are made from this 0.1 mg/L working standard

11.10.3 Preparation of the calibration standards. The low standard shall be at or below the reporting limit and the standards shall be in the linear range of the instrument, bracketing the expected sample concentration ranges. Follow the table below to make calibration standards

Table 11.1

Standard Concentration	Aliquot of 0.1 mg/L Working Standard	Reagent Water
0 µg/L	0 mL	100 mL
0.5 µg/L	0.5 mL	100 mL
3 µg/L	3.0 mL	100 mL
5 µg/L	5.0 mL	100 mL
8 µg/L	8.0 mL	100 mL
10 µg/L	10.0 mL	100 mL

11.10.4 The ICV and CCV shall be prepared at the midpoint calibration standard concentration. Use the table above to produce a 5.0 µg/L concentration from the primary initial solution.

11.10.5 The LCS level should be at a mid-level calibration standard (6.667 µg/L). The LCS is prepared by spiking 2.0 mL of the 0.1mg/L working standard into a calibrated 50-mL digestion tube. The volume is adjusted to 30 mL with reagent-grade water. The LCS is digested with field, MS/MSD, and MB samples.

11.10.6 The MS/MSD samples are at the same concentration as the LCS (6.667 µg/L). The MS/MSD is prepared by spiking 2.0 mL of the 0.1 mg/L working standard into duplicate liquid or oil samples (30 mL or smaller volume diluted to 30 mL), or into duplicate 0.3-g solid samples, before digestion

11.10.7 Secondary source standard shall be purchased from a different vendor, unless the primary vendor can supply different lot numbers.

11.10.7.1 The secondary source standard concentration is 1000 mg/L of mercury. A 1.0 mL aliquot of standard is diluted to 100mL in a volumetric flask with 5 mL conc. HNO_3 and the remainder with reagent-grade water to produce a 10.0 mg/L initial standard solution

11.10.8 The RS is prepared from the secondary source standard at a concentration near the midpoint of the calibration curve. Use the table above to produce a 5 µg/L concentration from the secondary standard solution

11.10.9 The method blank is prepared with each sample batch using reagent water equal to that used for the samples. The purpose of the method blank is to monitor potential reagent and/or laboratory contamination. The method blank is subjected to the same procedures as the samples

12.0 Sample Containers, Preservation, And Handling

12.1 All sample containers must be pre-washed with detergents, acids, and reagent water. Plastic and glass containers are both suitable.

12.2 Aqueous samples must be acidified to a pH of <2 with nitric acid. Holding times for mercury samples are 28 days.

12.3 Non-aqueous samples shall be refrigerated and analyzed within 28 days.

13.0 Quality Control

13.1 The analyst must demonstrate proficiency in performing the analysis. Method proficiency must be re-demonstrated anytime a major method modification is made, a major software revision is added, or a major instrument modification is made. Demonstration of method proficiency may also be required after major instrument maintenance. This is decided on a case-by-case basis through discussions with the Laboratory Technical Manager and Quality Assurance Manager.

13.2 An initial demonstration of instrument performance should be performed before analyzing samples. Performance of an MDL study should be done prior to using this method. MDL studies should be done annually, or when major changes are made to equipment or procedures.

13.3 A preparation batch of samples is defined as a group of up to twenty field samples of similar matrix type that have been prepared at the same time or time sequence with the same lots of reagents for the same analysis. In addition to the twenty samples, each preparatory batch will contain at a minimum, a method blank, a laboratory control sample, a matrix spike, a matrix spike duplicate and a matrix duplicate. Preparation batches of samples may be continuously strung together in these run sequences, as long as the analytical batch QC requirements meet the acceptance criteria established within the appropriate SOP.

13.4 An analytical or instrumental batch is defined as samples that are analyzed together within the same time period or in continuous sequential time periods. Within the analytical batch are included individual QC requirements as defined by the analytical (determinative) method.

13.5 Run a calibration curve on a daily basis that employs a minimum of a calibration blank and five standards. Acceptance criteria are presented in Section 18.

13.6 A second source standard shall be used to verify the calibration curve (RS). Acceptance criteria are presented in Section 18.

13.7 An ICB is analyzed following the ICV. Acceptance criteria are presented in Section 18.

13.8 The calibration must be continually verified using the CCV and the CCB. The CCV and CCB shall be run after every 10 samples and at the end of the analytical run. Acceptance criteria are presented in sections18.

13.9 A MB shall accompany each sample batch to determine if contamination or any memory effects are occurring. A batch consists of samples digested together using reagents from the same lots. A method blank is a volume of reagent water acidified with the same amounts of acids as were used for preparation of the standards and samples. This sample is carried through the entire digestion and analysis procedure. Acceptance criteria are presented in Section 18.

13.10 Control charts are maintained for LCS results. However, the acceptance criteria derived from these charts may not be appropriate if the charted ranges are too wide.

13.10.1 Accuracy control charts have a maximum default range. The maximum default range for CAS is 70-130%. Specific acceptance requirements are listed in section 18.

13.10.2 Precision control charts tracking RPD of interbatch LCSs should fall within the control limits. However, if RPD results are outside these control limits, the batch will not be rejected, as long as the LCS recovery is acceptable. This precision information should be evaluated to see if systematic problems could be identified. If problems are suspected, the method should be fully evaluated.

13.11 The use of the matrix spike and matrix spike duplicate will depend in part on what role the laboratory is playing. Acceptance requirements are located in section 18

13 11.1 When the laboratory serves the role as the primary laboratory, then site-specific documents should be consulted. The sample to be used for the MS/MSD may be specified in the field. This previously designated sample would then be spiked at a concentration equivalent to the site action level or lower. The MS/MSD would be prepared using the secondary source standards.

13.11.2 If this information was not specified or unknown, then the laboratory would chose a representative sample from each batch of samples analyzed.

13.11.3 If samples from multiple sites were to be analyzed in the same batch, then multiple sets of MS/MSDs may be required.

13 11.4 When the laboratory serves the role as the QA laboratory, the above scenario may not be practical to implement.

13.11.5 If the site-specific requirements are unknown and samples from multiple sites are analyzed in the same batch, then the laboratory should select a single sample for spiking from one of the projects. Each batch of samples would then contain at least one MS/MSD pair.

13.12 All sample analytical results used for final data reporting must be above the laboratory reporting limit (LRL) and below the high standard of the calibration curve. Data shall be checked to ascertain if it conforms to accepted practices for reporting of results at or near the MDL.

13 13 Method of Standard Additions - The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards It will not correct for additive interferences that cause a baseline shift

13.13.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution are taken. To the first (labeled A) is added a known volume of a standard analyte solution of a specific concentration. To the second aliquot (labeled B) is added the same volume of solvent The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration is then calculated; see section 10 3 for equation. The volume and concentration of the standard solution should be chosen to avoid excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

13 13.2 Improved results can be obtained by employing a series of standard additions. Two equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume For example, addition 1 should be prepared so that the resulting concentration is approximately 50% of the expected absorbance of the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100% and 150% of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. A linear regression program may be used to obtain the intercept concentration

13.13.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

13.13.3.1 The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised

13.13.3.2 The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte. The determination must be free of spectral interferences and corrected for nonspecific background interference

14.0 Calibration and Standardization

14.1 A calibration curve is prepared using a six point calibration curve, which includes the calibration blank. Standards are evaluated from lowest concentration to highest concentration

14.2 The linearity of the curve is evaluated prior to proceeding with analysis. See section 18 for acceptance criteria.

14.3 The following standards are used for the calibration curve:

Table 14.1

Standard	Concentration
#1	Calibration Blank
#2	0.5 ug/L
#3	3.0 ug/L
#4	5.0 ug/L
#5	8.0 ug/L
#6	10.0 ug/L

14.4 For further details on calibrating the instrument, refer to Section 15.0

15.0 Procedure

15.1 For solid samples:

15.1.1 Add 0.3-0.4 grams of sample and 5 mL of reagent water to a digestion tube. This should be done in triplicate for each sample. MB, LCS/LCSD, and MS/MSD samples are also prepared for digestion. Calibration standards, ICB, ICV, CCB, CCV, and RS standards are not digested, but all the following reagents are added.

15.1.2 Add 1 mL of nitric acid and 2.5 mL of sulfuric acid to the digestion tube.

15.1.3 Add 2 mL of potassium persulfate solution followed by 5 mL of potassium permanganate

15.1.4 Per batch of 10 samples or less there should be at least one MS/MSD sample; for batches greater than 10, there must be an MS/MSD every 10 samples.

NOTE: If the solution is purple after one hour, sufficient permanganate was added. However, it the solution is clear before an hour, the permanganate is being used up by interference. Add more

permanganate to maintain the purple color for one hour during the digestion period. The same amount of permanganate must be added to all samples.

15.1.5 Tighten lids and then shake.

15.1.6 Mix and heat in a hot block for approximately 2 hours at 95°C.

15.1.7 At the end of 2 hours, remove the tubes from the hot block and allow them to cool for one hour.

15.1.8 Add 5 mL of hydroxylamine sulfate to the solution and swirl immediately to remove purple color.

15.1.9 Allow solution to settle to clearness before analyzing the sample. Analysis should be performed as quickly after digestion as possible.

15.1.10 After settling, add 25 mL of DI water.

15.2 For the liquid samples:

15.2.1 Add 30 mL of the sample and put into a digestion tube. MB, LCS/LCSD, and MS/MSD samples are also prepared for digestion. Calibration standards, ICB, ICV, CCB, CCV, and RS standards are not digested, but all the following reagents are added.

15.2.2 Add 1 mL of nitric acid and 2 mL of sulfuric acid to the digestion tube.

15.2.3 Add 2 mL of potassium persulfate solution followed by 5 mL of potassium permanganate.

15.2.4 Per batch of 10 samples or less there should be at least one MS/MSD sample; for batches greater than 10, there must be an MS/MSD every 10 samples.

NOTE: If the solution is purple after one hour, sufficient permanganate was added. However, it the solution is clear before an hour, the permanganate is being used up by interference. Add more permanganate to maintain the purple color for one hour during the digestion period. The same amount of permanganate must be added to all samples.

15.2.5 Tighten lids and then shake.

15.2.6 Heat in a hot block for approximately 2 hours at 95°C.

15.2.7 At the end of 2 hours, remove the digestion tubes from the hot block and allow them to cool for one hour.

15.2.8 Add 2 mL of hydroxylamine sulfate to the solution and swirl immediately to remove purple color.

15.2.9 Allow solution to settle before analysis. Sample analysis should be performed as quickly after digestion as possible.

15.3 Set up instrumentation and hydride generation apparatus.

15.3.1 Fill reagent containers.

15.3.2 Turn on the nitrogen carrier gas.

15.3.3 Place reagent pump tubes on the peristaltic pump, and lock down with the pump clamp.

15.3.4 Turn on the VGA-76 pump. Check the low gas pressure light; if the light is off, nitrogen carrier gas flow is sufficient. If the light is on, check the nitrogen gas supply.

15.3.5 Adjust the screws on the pump clamp to adjust the reagent flow. Tighten the screws sufficiently so both reagent lines and the sample line are flowing smoothly and continuously with no backflow. Overtightening the screws can cause premature wearing of pump tubing and strain the peristaltic pump.

15.3.6 Gas and reagent flow rates into the instrument are set internally and do not need to be adjusted.

15.3.7 Set wavelength on the AA to 253.7-nm and the slit width to 0.5-nm (or manufacturer's recommended slit-width).

15.3.8 Allow a minimum of fifteen minutes for the lamp and instrument to warm up.

15.4 Start the program to begin calibration; after the program loads, you will be prompted to present the next solution for analysis.

15.5 Place the sample feed line into the calibration blank; allow the sample to flow into the system before pressing "Read" to begin reading the sample.

15.6 After the calibration blank, run calibration standards from low to high to obtain a calibration curve. Evaluate the linearity of the calibration curve. The acceptance criterion is stated in section 18.0.

15.7 Following an acceptable calibration, run the ICB, ICV, and RS standards. These must meet the requirements of Section 18.0.

15.8 Set up analytical run; if data system automates the analytical run, program samples into software.

15.9 Run the MB and LCS before starting samples. Every batch of 10 samples should include an MS/MSD; use the following table as a guide to setting up an analytical run.

15.10 Continuing calibration verification must follow the requirements found in Section 18.0.

15.10.1 During the analyses, the MS, LCS, and method blank samples need to be evaluated. MS sample acceptance criteria are located in Section 18.0. If the MS samples indicate a matrix effect, the method of standard additions needs to be considered.

15.10.2 An LCS must be analyzed with each sample batch. The mercury concentration level should be near the mid-level standard. Control charts will be maintained for the LCS. Acceptance criteria are located in Section 18.0.

15.11 Method blank requirements are found in Section 18.0.

Note: Project DQOs take precedent over these requirements.

16.0 Calculations

16.1 The concentration of mercury (µg/L) in the samples is read from the calibration curve or directly from the instrument if operating in the direct concentration mode. Samples that required dilution are adjusted according to the following equation:

$$\text{mercury } (\mu g/L) = A \times \left(\frac{B}{C}\right)$$

where:

A = sample concentration from calibration curve
B = volume (mL) of calibration blank used to dilute sample
C = volume (mL) of sample used

16.2 The concentration of mercury in solid matrices is reported in mg/kg of waste as follows:

mg/kg of mercury = A(V/W)

where:

A = final concentration read from calibration curve in µg/L
V = Final volume of processed sample (L) (0.1 L recommended)
W = Dry weight of sample digested (g)

16.3 Calculation of Recovery for MS/MSD and LCS analyses

$$\%\text{Recovery} = \frac{C_s - C_u}{C_n} \times 100$$

where:

C_s = Measured concentration of the spiked sample aliquot
C_u = Measured concentration of the unspiked sample aliquot (use 0 for LCS analyses)
C_n = Nominal (or theoretical) concentration of the spiked sample aliquot

16.4 Calculation of Precision

$$\%\text{RPD} = \frac{(C_1 - C_2)}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where:

C_1 = Measured concentration of the first sample
C_2 = Measured concentration of the second sample

16.5 Correlation coefficient

$$R = \frac{\sum XY}{\sqrt{\sum X^2 \sum Y^2}}$$

where:
R = correlation coefficient
X = standard concentration
Y = absorbance

17.0 Method Performance

17.1 See QA/QC documentation for the latest MDL study results and method performance information

17.2 See method 245.1, EMMC Version, Section 13.0 and Table 1 for method performance data.

18.0 Data Assessment and Corrective Actions

Table 18.1 QC Acceptance Criteria

Standard	Acceptance Criteria		
	SW846 7470/7471	EPA 245.1	CLP
Instrument Blank (initial)	Zero	Zero	Zero
Calibration Curve	r-value = 0.995 or greater	r-value = 0 995 or greater	r-value = 0.995 or greater
ICV	%Recovery = 90-110%	%Recovery = 90-110%	%Recovery = 80-120%
ICB	Below reporting limit	Below reporting limit	Below reporting limit
RS	%Recovery = 90-110%		%Recovery = 80-120%
MB	Below reporting limit	Below reporting limit	Below reporting limit
LCS			%Recovery = 80-120%
MS/MSD	%Recovery = 80-120% 50-150% TCLP	%Recovery = 85-115% (optional)	%Recovery = 75-125%
MS/MSD	N/A	N/A	RPD = ≤ 20%
Duplicates	RPD = ≤ 20%	RPD = ≤ 20%	RPD = ≤ 20%
CCV	%Recovery = 80-120%	%Recovery = 90-110%	%Recovery = 80-120%
CCB	Below reporting limit	Below reporting limit	Below reporting limit

18.1 The correlation coefficient of the calibration curve must be calculated and must be ≥0 995. If not, recalibration must be performed.

18.2 The ICV measured concentration limits varies per method. See Table 18.1 for applicable limits. If acceptance criteria are not met, recheck standard curve and ICV preparation and/or perform routine instrument maintenance (e g., replace tubing).

18.3 The ICB must be less than the MDL. If greater than MDL, recheck standard curve and ICB preparation and/or perform routine instrument maintenance (e.g , replace tubing).

18.4 The acceptance limits of the CCV standard varies per method. See Table 18.1 for applicable limits. If not, terminate the analysis, correct the problem, and reanalyze the samples back to the last CCV standard within QC requirement.

18.5 The results of the CCB must be less than the reporting limit. See Section 3.0 for current reporting limits. If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples

18.6 The analyst shall confirm that these blanks were analyzed at the required frequency.

18.6.1 The method blank should not exhibit any contamination of any analyte above the reporting limit for any of the method target analytes. Corrective action should be performed any time method target analytes are detected above the reporting limits to reduce and control contamination. Current reporting limits are found in Section 3.0.

18.7 Corrective action will be required if site-specific target analytes are detected at greater than 5% of the regulatory limit for that analyte or if the concentration in the blank is greater than 5% of that in the sample.

18.7.1.1 Assess the effect of the contaminant on the samples. Corrective action would include reanalysis of field and QC samples in the batch if some or all of the samples also contained levels of target analytes that exceeded the above criteria.

18.7.1.2 If none of the field samples had values above the stated criteria, then reanalysis may not be necessary.

18.7.1.3 The source of contamination should still be investigated and reduced/eliminated. Anytime contamination is noted in the method blank, the situation and impact on the data should be discussed in the case narrative

18.8 Assess that LCS was prepared at the required frequency. Plot the target analyte on appropriate control charts. If accuracy is not within control limits, reanalyze the LCS digestate. If still unacceptable, re-digest and reanalyze a new LCS and all associated samples. If the LCS is still unacceptable, the entire procedure (digestion and analytical) must be systematically investigated to locate the source of the error. See Table 18.1 for LCS limits.

18.9 Assess that matrix spike/matrix spike duplicates were analyzed at required frequency. The analyst shall also verify that the samples were spiked at the appropriate level. The order of preference for spiking levels is as follows:

18.9.1 If the target analyte concentrations are known, spike to increase the background concentration by a factor of approximately two

18.9.2 If an action level exists, spike at this level

18.9.3 If neither of the first two conditions applies, spike at a level that corresponds between the low and mid-level calibration standards.

18.10 Acceptance criteria for percent recovery and/or RPD results must meet project-established goals, if available, or method requirements. If no project goals are specified or there are no method requirements, then results must be within the indicated control limits on the appropriate control charts or the maximum default ranges, 70-130% and RPD <20. See Table 18.1 for method specific limits. If these conditions are not met, the MS/MSD sample digestates must be reanalyzed to verify a matrix effect. If the results are still unacceptable, the analyst shall notify the Laboratory Technical Manager and Quality Assurance Manager to determine appropriate corrective action. MS/MSDs are not used to determine the acceptability of a digestion batch, yet the client must be notified of unacceptable results in the case narrative.

18.11 If both LCS and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be re-digested and re-analyzed.

18.12 If the MS/MSD is unacceptable, but the LCS is acceptable, then a potential matrix effect has been identified. The project manager must be contacted to discuss further alternatives and the potential impact on the project. Further alternatives may include re-digestion and reanalysis. Reasonable attempts must be made to address the matrix interference by using the method of standard additions, a different matrix modifier, or an equivalent approach.

18.13 Assess matrix duplicates were analyzed at required frequency. Acceptance criteria are that all RPD results meet project-established goals. If no project goals are specified, then the results must be within the indicated control limits on the appropriate LCS precision control charts or the maximum default range of < 20 RPD. If these conditions are not met, perform the following corrective actions as appropriate:

18.13.1 Reanalyze the sample and duplicate digestates to verify a matrix effect.

18.13.2 If the duplicate precision is still unacceptable, then a potential matrix effect has been identified. The project manager must be contacted to discuss further alternatives and the potential impact on the project. Further alternatives may include re-digestion and reanalysis.

18.14 The RPD between duplicate injections for each sample must be <20% for concentrations above the low standard. If not, reanalyze. If still unacceptable, perform instrument maintenance as needed and reanalyze.

18.15 The analyst must verify all reported results are derived from analytical results that are either above the LRL or below the highest standard of the initial calibration curve. Verify the results are reported as follows:

18.15.1 Sample concentrations that have been analyzed using the digestate in its most concentrated form, which are not detected, should be reported as less than the LRL.

18.16 For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot. Verify that results have taken dilutions (or concentrations) into account.

19.0 Contingencies for Out-of-Control or Unacceptable Data

19.1 Most analytical data has been checked for acceptability during analysis. Analysts should flag any unacceptable data, make any relevant notes as to the cause, or record any observations noted that may assist in the evaluation of the data.

19.2 Some QA/QC parameters may not be required for a certain method. The laboratory is often running additional QC standards beyond standard requirements. QC should meet the minimum required by the reported method.

19.3 If QC biases high, and samples are below detection limits, results can be reported.

19.4 If an analyst cannot solve QA/QC problems, an irregularity report should be filled out and given to QA/QC for evaluation. If instrument problems persist, maintenance and troubleshooting should be performed to fix the problem.

20.0 Waste Management and Pollution Prevention

20.1 This procedure generates acidic wastes. All wastes shall be disposed of according to local regulatory as well as laboratory-established guidelines. Refer to the Laboratory's Disposal SOP A-2050.

20.2 Pollution prevention is any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. This procedure generates acidic waste and relies on concentrated metals standards for preparation. The laboratory should minimize the amount of acids used whenever possible. Small volumes of standards are used to create calibration and QC standards. Concentrated metals standards should be ordered in quantities sufficient to be fully utilized by their expiration date, to eliminate unnecessary disposal of concentrated metals standards.

21.0 References

21.1 *Method 245.1, Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry* Revision 3.0 EMMC Version, 1994.

21.2 *Test Methods for Evaluating Solid Wastes, SW-846*, Third Edition, Update III, July 1992, Method 7470A.

21.3 Sample Waste Disposal, A-2050.

21.4 *Ohio EPA Laboratory Manual for Chemical Analyses of Public Drinking Water*, 2000.

21.5 *Guide to Environmental Analytical Methods*, Edited by Robert E. Wagner and William Kotas, Genium Publishing Corporation, 2nd. Edition 1992

21.6 *Standard Methods for the Examination of Water and Wastewater18th Edition,* Edited by Arnold E. Greenburg, Lenore S. Clesceri, and Andrew D. Eaton, American Public Health Association, 1992

21.7 Online National Environmental Methods Index, www.nemi.gov, July 2004.

21.8 *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, Office of Superfund Remediation and Technology Innovation USEPA, OSWER 9240.1-45, EPA 540-R-04-004, October 2004

Appendix A. Drinking Water Testing Requirements

Section 1.01 The following procedures are followed when analyzing drinking water samples for mercury; these procedures are additions or modifications to normal liquid sample procedures

(a) A reporting limit check (RLC) must be analyzed at the beginning of the analytical run. It should be analyzed immediately following the ICB and ICV

(i) Prepared concentration should be 0.2 ug/L. Prepare from the 0.1 mg/L working standard.

(ii) Acceptance criteria is ± 30%. If the RLC fails, prepare a second RLC and check; if the second check fails, perform an instrument recalibration.

(b) Two RS standards are run following the RLC to validate the calibration curve. The RS standards are made from a secondary standard source; one is a low-range (at or below 10 times the reporting limit) and the other is a high range (at or above the highest response used for samples). Acceptance criteria is 90-110%.

(i) Prepared concentration for the low-range should be 2.0 ug/L.

(ii) Prepared concentration for the high-range should be 10.0 ug/L.

(c) Run samples in the following order: MB, LCS, sample A, sample B, etc., including duplicate samples and MS/MSD samples at frequency of one in ten or fewer samples. If the spike recovery or RPD is out of the acceptable range, the sample must be diluted and spiked. If it fails again, the sample must be analyzed by the methods of standard additions.

(d) After each ten analyses (spikes count as one analysis each), run a CCV, CCB, and RS (low or high). If the CCV and RS do not meet acceptance criteria, terminate the sequence, correct the problem, recalibrate and re-analyze the samples and spikes between the last acceptable CCV or RS and the failed CCV or RS

(e) When all samples and spikes are analyzed, re-analyze the CCB, CCV, and RS (low or high)

(f) At least once every three months a minimum of one actual drinking water sample must be analyzed to maintain certification. If no sample has come in, analyze a tap water sample from the lab.

Table A1: QC Acceptance Criteria for Drinking Water

Standard	Acceptance Criteria
Instrument Blank (initial)	Zero
Calibration Curve	r-value = 0.995 or greater
ICV	%Recovery = 95-105%
ICB	Below reporting limit
RLC	%Recovery = 70-130%
RS	%Recovery = 90-110%
MB	Below reporting limit
LCS	%Recovery = 85-115%
MS/MSD	%Recovery = 85-115%, 70-130% if BDL
MS/MSD	RPD = ≤ 20%
Duplicates	RPD = ≤ 20%
CCV	%Recovery = 90-110%
CCB	Below reporting limit